VOLUME 4 MAY 1994

THE *SEM* INAR

The newsletter that addresses the *probing* concerns of Forensic Science

PEAK PERFORMANCE #1

This is the first of what I hope will be a helpful and productive series of columns covering the topic of energy-dispersive spectrometry (EDS) and its applications. If, upon first glance, EDS appears to be based on a complicated series of intimately related interactions requiring in-depth knowledge of a wide range of topics, that's only because it is. With a little luck and perseverance, however, I hope we can de-mystify such daunting topics as spectrum statistics, beam-specimen interactions, quantification (which may take a few trys), X-ray mapping, and others. If you have any questions, complaints, or would like to suggest topics for me to address, please fax them to me at the Tarrant County Medical Examiner's Crime Laboratory, Fort Worth, TX, (817) 927-0902.

To give you a bit of background as to why I'm doing this, I spent a year at Michigan State University, after graduating with my Master's, at the Center for Electron Optics running a wide variety of customer samples, I got a job at Link Analytical (now Oxford Instruments Microanalysis Group) as an Application Specialist. For four years, I trained people of all levels of knowledge, from people who couldn't spell "S-E-M" to Ph.D.'s, how to run microanalysis systems

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Editors:
Max Houck, Tarrant County Medical Examiners Crime Laboratory, Fort Worth, TX
Dennis Ward, FBI, Washington, DC

AAFS MEETING

One of the most interesting and thought provoking talks at the recent American Academy of Forensic Sciences Meeting in San Antonio was the presentation by **Robert H. Schwartz** of the McCrone Research Institute entitled "The Analysis of Gunshot Residue Particles Collected from the Nasal Cavity". Mr. Schwartz described his recent experiences in developing a method of collecting, isolating, and identifying gsr particles entrained in the nasal mucus of a person involved in firearms discharge. He described in a highly entertaining manner the trials he went through to find a method of blowing a test subject's nose on a sheet of cellophane and the difficulties inherent in going 48 hours without blowing or picking your nose. It was found that collection on cellophane followed by separation of the gsr particles from the mucus in a hot water bath and suitable filtration produced samples on which gsr particles were readily identified using SEM-EDS. Preliminary studies indicate that hundreds of particles are trapped by the nose of a shooter after only a single shot and these are readily detectable until the nose is blown repeatedly to remove the mucus and particle mixture. In one

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and helping them solve sample analysis problems. I developed an intense love of the methodology and teaching/helping people, who either wanted to or had to, also love it. Currently, I am a trace analyst and forensic anthropologist for the ME's Office Crime Lab, among other responsibilities. Dennis made me say all this.

As a matter of convention, technical terms which are introduced will be **bolded** and followed by a definition in [brackets], if the meaning of the term is not obvious from its context in the sentence. Unless otherwise noted, the only reference I will use in this column will be *Scanning Electron Microscopy and X-Ray Microanalysis*, 2nd ed., Goldstein, J.I., Newbury, D.E., Echlin, P., Joy, D.C., Romig, Jr., A.D., Lyman, C.E., Fiori, C. and Lifshin, E., 1992, Plenum Press: New York. This book is the Rosetta Stone for understanding and deciphering the luxuriant mysteries of x-ray microanalysis; my hope is that this column will take away the fear the novice user might have in approaching "Goldstein" for further information.

To begin at the beginning, let's look at the source and nature of the signals with which we are concerned. While viewing a sample with an SEM may be considered, and often is, the purpose of the instrument, taking such a limited approach would ignore the possible exploitation of analytical signals that are generated as a by-product of exposing a sample to an electron beam. EDS capitalizes on the "soft" X-rays generated (those with wavelengths between 10^{-9}m and 10^{-11}m) when the electron beam excites the atoms in a sample; in fact, its physical principles are similar to X-ray fluorescence (XRF) except that the electron beam takes the place of the primary X-ray tube. When a **primary electron** [a beam electron which has not interacted with the sample] interacts with an atom of the sample, it can dislodge an inner shell electron (Figure 1). Since electrons always seek the lowest possible energy shell, the ejection of an inner shell electron results in its replacement by an electron from a higher order shell. The energy the higher order electron "gives up" in this electron jump is equal to the difference in energy between the two shells; this energy is released in the form of a photon, which registers with the EDS detector as one X-ray count of that particular energy. For a specimen electron to be removed, the primary electron's energy must exceed the energy binding the sample electron to it's shell (the **binding energy**) and, therefore, the necessary energy to eject an electron varies from shell to shell and element to element.

Nomenclature for X-ray peaks is generated by the shell from which the ejected electron originated (K, L or M) and the number of shells "jumped" to fill that vacancy, where one jump is referred to as á, two jumps as â and three jumps as ã; this is the Siegbahn notation system which is different from the system used in atomic physics. So, an iron Ká peak is generated by energy released from the ejection of electrons from the K shell and filled by electrons from an adjacent shell, one level away. If sufficient energy exists in the primary beam to excite K X-rays, L and M X-rays will also be produced, if these shells are occupied. Additionally, not all of the electrons in each shell possess exactly the same energy and variations will occur; for example, the Fe Ká peak is actually made up of two peaks, $Ká^1$ and $Ká^2$, which cannot be distinguished by the EDS detector and are displayed as one slightly larger peak. This is why higher atomic number elements, with more electrons per shell, produce such complicated peak arrays, particularly from the L and M shells.

The X-rays will have discrete energies which can be expressed in electron volts (eV) or thousands of electron volts (keV); like visible light photons, X-ray photons have a wavelength which is related to their energy:

$$ë = hc/E \text{ nm}$$

where *h* is the Planck constant, *c* the speed of light, *E* the energy in keV and the result is given in nanometers (nm).

The specific energy required to excite any given X-ray line is called the **absorption edge** energy or the **critical excitation** energy. This value is always slightly larger than the actual emission line. Therefore, the binding energy of an electron must be exceeded by 2-3 times its energy

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to efficiently produce X-rays. For example, if the FeKá peak appears at 6.398 keV, we must set the accelerating voltage at about 15 kV to excite this peak. These differences in defining, or **characteristic**, X-ray energies allow the detection and identification of elements from beryllium on up the periodic table. The energy of the characteristic X-ray radiation for a given series of lines varies monotonically with atomic number [**Moseley's Law**]:

$$\ddot{e} = B/(Z - C)^2$$

where ë is the characteristic X-ray wavelength, Z is the atomic number of the element in question and B and C are constants which differ for each "family" of lines, i.e., that element. The upshot of this equation is that if the energy of a particular line (K,L,M) is measured, then the atomic number of the element producing that line can be determined. This relationship can be shown graphically as in Figure 1.

If a primary electron has insufficient energy to dislodge a specimen electron, its deceleration results in the production of non-characteristic X-rays: This constitutes the **background** or **bremsstrahlung** [German, "breaking, decelerating radiation"] signal of an EDS spectrum.

Next time, unless anyone has a better idea, I'll speak about how discrete X-ray lines end up being broad peaks.

This column, and the entire series, is dedicated to the memory of Chuck Fiori, for obvious reasons.

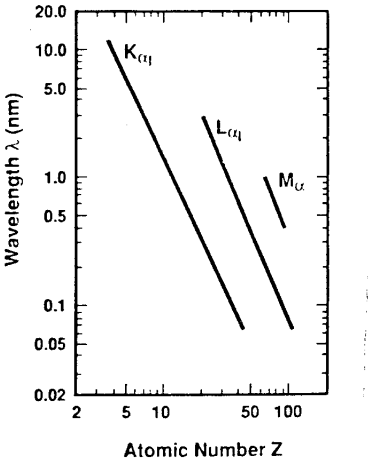

Figure 1: Moseley's relation between ë and Z for the Ká1, Lá1 and Má1 characteristic X-ray lines (from Goldstein, et al., 1992)

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controlled test, over 400 gsr particles were found in a sample collected after 24 hours from the time of firearm discharge despite the fact that the subject blew his nose one time during the waiting period. The large number of particles found and the long time intervals possible when the nose is left undisturbed look promising for the future of this technique. Bob promises to have the results of further practical studies involving a variety of firearms and activity scenarios ready to present at the Inter/Micro-94 meeting in July.

..contributed by Robert Koons, FBI/ FSRTC, Quantico, VA

additionally. . .

K. Furukawa and **T. Shinohara**, in "Discrimination Between Glass Samples of a Foglamp by Elemental Analysis Using Electron Probe Microanalyzer" studied fog lamp glasses by EPMA, comparing samples by multivariate analysis of the ratios of the primary oxides. **Ronald Grier** et al in "Bite Marks in a Child Abuse/Murder" used EM to show individual characteristics of marks in bite mark impressions. **Max Houck,** in "A Survey of Gunshot Residue Analysis Methods", reviewed aspects of GSR analysis, including standard procedures, collection methods, thresholding problems and specificity of the data in US and Canadian labs from survey results of these labs. And **Thomas Hopen** and **John Wueper** in "Pyramids in the Sands" used LM and SEM techniques to characterize minerals for their geological significance. See micrograph on p 15.

TRICKS, TIPS

Consider using *pyrolytic* carbon planchets for SEM specimen mounts. With a glass-like non-porous surface, they provide a structureless background for LM and EM observation/photography. A uniform, thin adhesive layer can easily be applied by cover slip/capillary dispersion of the carrier fluid. Expensive, but reusable.

. *..Dennis Ward, FBI*

In the last issue of THE *SEM*INAR, **Scott Ryland** asked the community for assistance with the definition of structure of several paint samples encountered during casework. In response, **Dr. W. Stoecklein, Wolfgang Langer,** and **Rainer Goebel** of the BKA queried their "Forensic Vehicle Paint Collection" and faxed their answer to Scott. We appreciate their outstanding effort to share their expertise with the community. Below is a copy of their response.

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RYLAND RESPONSE

consists of all coating materials like zinc coatings, phosphate coatings, first primers, chip resistant primers, primer surfacers and top coats (solid colors, metallic - paints, pearlescent laquers and clear coats) applied on makes and models sold on the German market since about 1980. This is to say that we deal with the coating material used by the Japanese, French, Spanish, English, Italien, Swedish, Finnish and German car manufacturers as far as it concerns the German market.
The metallic layer you came across in your casework is one among several anti-corrosive measures, consisting of a metall layer, to provide improved corrosion prevention . The measures used depend on the make, model and year of production.

Normally most car manufacturers are using zinc galvanized steel sheets for the protection of the body parts which are especially exposed to the impact of stones or chips and therefore are most susceptible to corrosion . Those parts are for example the front panel, the hood and the front fenders.
But some car manufacturers like Audi or Porsche are also employing bodies which are completly galvanized with zinc.

Other car producers are using more sophisticated systems.

To start with , Peugeot and Toyota are using steel sheets which are precoated with an iron - zinc alloy and which are called, in the case of Toyota, Excelite - sheets. But we have also learnt that if there is a special heat treatment of the conventional zinc galvanized sheets, a more or less homogeneous distribution of iron, which is diffused from the steel surface into the zinc coating , could be detected .

Another measure to improve repairability and corrosion resistance is applied by Nissan. They use a new type of anti-corrosive precoated steel sheets instead of the conventional zinc-coated steel sheets. This so called durasteel is an electroplated, zinc-nickel alloy under an organic film and is produced as one-side precoated steel as well as two-side precoated steel. Especially the two-side precoated steel should provide better corrosion resistance because it protects the inside as well as the outside of the body.
Such durasteel sheets could be found in use for the following body component parts depending of the model: front panel, front hood. front fender, front door outer panel , rear door outer panel , slide door , rear fender, back door, trunk panel.

A similar zinc - nickel precoating is also used for body parts of the Subaru model Libero.

Whereas the above mentioned anti-corrosive measures consist of pure metal layers, FORD and OPEL are applying systems which additionally contain organic components.

Opel occasionally applies a zinc dust primer on such body parts as the frame door opening or in and around the openings of the headlights.

In 1988 ,Ford Germany started to employ the so called Duplex -sheets beginning with the model Scorpio , followed in 1989 with the model Fiesta . These precoated steel sheets show the following succession of layers:

First , next to the steel surface, is an electrolytic coat consisting of zinc or zinc-nickel alloy. This is followed by a zinc phosphate coating which is covered with zinc dust and/or aluminium flakes containing organic binder which is based on epoxy resin.

The capability of the above mentioned anti-corrosive systems is based on the difference of iron and zinc in the electrochemical series of the elements. Zinc is less precious than iron , so in the case of corrosive conditions zinc will act as the anode, that is to say it will react to form white rust (zinc oxide / zinc hydroxide) while iron as the cathode will stay inert.

Referring to the 1991 GEO Metro, we would like to call your attention to the fact, that the GEO Metro is the export version of the Suzuki model Swift for the North American market. The version for the German market consists of zinc galvanized body parts including the hatch back, which could show a zinc - iron containing metal layer below the first primer.

If you have any further questions please contact the following address.

Bundeskriminalamt
Kriminaltechnik 12
Dr. Stoecklein / W.Langer
Thaerstraße 11

D- 65193 Wiesbaden

Yours sincerely

Dr. W. Stoecklein

Wolfgang Langer

Robert S. White has offered forensic analytical/consulting/expert witness services since retiring from the West Virginia State Forensic Crime Laboratory in 1992. Now, R.S. White has joined forces with CamScan USA Inc. to offer Gunshot Residue Analysis using state-of-the-art SEM/EDX instrumentation. General trace analysis and investigation using scanning electron microscopy and energy-dispersive xray analysis is also available. For more information, please contact:

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W. Va. State Police (Retired)
7006 Kanawha Farm Circle
Charleston, WV 25312-9417
(304) 984-9456

or

Tony Owens
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(412) 772-7434 Fax.

note: obviously we missed publishing this announcement on a timely basis, but included it for "historical" and general interest purposes.

The Southwestern and Southern Associations of Forensic Scientists are holding a combined meeting April 12-16 at the Little Rock Hilton Inn, Little Rock, Arkansas

Two full days (Wed and Thurs) of SEM/EDX workshop are scheduled. Max Houck (Tarrant Co Medical Examiners Office, Texas) will demonstrate SEM/EDX for GSR and paint chip analysis. The workshop will be hosted by Zeiss and Oxford Instruments. Those of you who know Max from his former life as Oxford Applications Specialist are aware of his expertise and excellent teaching ability. The second day is a hands-on for those who wish to bring their own “special sample” requiring special attention. Class size, naturally, is limited.

For further information, call Gary Lawrence, Ken Michau, or Nick Dawson at 501/227-5747.

A *GSR (gunshot residue) symposium/roundtable discussion* will be held at the joint meeting of the Canadian Society of Forensic Science (CSFS) and Northwest Association of Forensic Scientists (NWAFS), Oct 31-Nov 5, 1994, Vancouver, B.C., Canada. Even if you can't attend, they would appreciate any suggestions you may have for discussion topics and/or problems for the planned roundtable discussion. Also, they are soliciting any unpublished research/observations regarding GSR, even if informal. This information will be bound, distributed at the meeting and made available to the community. Frank will summarize this meeting in a future issue of THE *SEM*INAR. If you can help, please contact Frank Boshears (206) 593-2006, or, for general information about the meeting, Jeff Coughlin (604) 264-3507.

note: This study was performed by the authors in 1989, and presented at the 1989 SAFS meeting in Raleigh NC. This work was not published elsewhere.

DEPOSITION OF GUNSHOT RESIDUE AT VARIOUS DISTANCES FROM DISCHARGING FIREARMS

ROBERT S. WHITE & MARK L. GROSS

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INTRODUCTION

Several analysts in the Gunshot Residue field (including workers at the West Virginia State Police Laboratory) had noticed the presence of large numbers of round spheroidal Pb particles on shooting victims usually with the absence of Pb-Sb, Pb-Ba or Pb-Sb-Ba particles. Also, just how far Gunshot Residue could possibly be deposited from a discharged firearm was questioned. These two areas provided the original impetus for the collection of experimental data.

Hopefully, this study will enhance the analysts knowledge of where one may expect to find Gunshot Residue deposited. This paper should increase ones understanding of what types of particles one may expect to find downrange and the importance of particle ratios.

EXPERIMENTAL METHODS

Firearms were discharged in an indoor and outdoor range at varying distances. The firearms were discharged into the middle of a nine inch by nine inch 100% cotton wipe which is suitable for clean room use. A new wipe was used for each different distance. Three blank wipes were sampled at random to ensure the cleanliness of the wipes.

After firing into the center of each wipe, the wipe was sampled with a polypropylene 3/4" diameter disc coated with 10% vistinex in trichloroethylene at least l inch from the bullet entrance. The samples were then concentrated in a manner taken from Loren Sugarman of the Orange County, Laboratory, in California.1

The samples were deposited on an 8 mm diameter nucleopore polyester membrane filter with .4um pores. The samples were placed on 3/4" carbon stubs and “painted” down with carbon dag. The samples were then carbon coated and ready for examination in the SEM.

The samples were analyzed on a CamScan Series 2 SEM. An accelerating voltage of 25 KV was used to ensure excitation of the Lead L line at 10.55 KeV. The working distance used for all analyses was 31 mm with a sample tilt of 20 degrees. An emission current of 10 microamps was employed with a standard tungsten hairpin filament.

A Tracor Northern 5500 EDS system was used. The resolution of the solid state detector is 147 ev/channel. The amplifier count rate was run in the high mode. The multi-channel analyzer was set for 2048 channels at .010 Kev per channel.

All analyses were run manually. A simple method of starting at the top left of the filter and going across to the right, then stepping down one frame and starting from the right back to the left was employed.

The firearms used to collect data for the distance determination were picked because of their

prevalence in cases submitted to the West Virginia State Police laboratory for Gunshot Residue determination. In 1987, 1988 and through February 1989 there were 239 different firearms (24 different calibers and types) used in various offenses. In some cases it was not possible to determine what type of firearm was used. However, the most frequently utilized firearms were 22 handguns and rifles, 25 automatics, 38 handguns and shotguns. The firearms used for data collection were: Smith and Wesson model 60 2" 38 caliber handgun; Smith and Wesson model 645 45 caliber handgun; Rossi 4" 22 caliber handgun; Titan 25 automatic 3 1/2" 1903 Springfield 30-06 bolt action rifle; and a Winchester Ranger 12 gauge shotgun 28". Of the 239 different firearms used in cases, 177 (74%) of them were the firearms picked to collect data.

All firearms were fired at a 9" x 9" cotton wipe at distances of 5', 8', 11' and 14'. Also, the Smith and Wesson 38 was fired at distances of 17' and 20'. The 30-06 was fired at 17'. The 38 was fired with the 9" x 9" wipes placed to the right, left and behind the weapon at distances of 4', 6' and 8'. The 30-06, 22 and the 38 were fired outdoors. The number of particles on each sample was approximated as was the particle density per square millimeter. When concentrated, the sample deposits in an area approximately 5 mm in diameter. This yields a surface area of 19.6 mm on the filter. The particle densities are in particles/mm.

EXPERIMENTAL DATA AND DISCUSSION

The ammunition used for data collection was all Federal. The breakdown of the ammunition used is as follows:

30-06 - 125 grain core-lokt
38 - 158 gr. Monark Round Nose Lead
45 - 185 gr. Monark Wad Cutter
22 - 40 gr. Hi-Power solid point
25 - 50 gr. Federal Full Metal Jacket
12 ga. - 5 1/2 shot 2 3/4" Magnum

The experimental data is contained in its entirety in Table I to allow a quick summary. Tables II, III and IV contain the information broken down into related areas. Table II contains information on firearms discharged on an indoor range with the samples collected downrange from the muzzle. Table III contains information taken from an outside range both down range from the muzzle and to the sides and back of the 38. Table IV contains information on samples taken from the sides and back. The percentage of Pb-Ba-Sb particles in Table II and III is fairly consistent. However, Table IV is out of the range of the other two. Also, the percentage of Pb particles is similar for Table II and IV while Table III is out of the range. In Table III, the amount of sample was generally much lower than in Table II and Table IV. The wind during the outside shootings was 5-10 mph and was obviously enough to blow away much of the sample. Also, some of the samples had problems in preparation and most of the sample was lost. An example is the shotgun at 11', the 22 inside at 8' and the 38 6' left. (This also points out the compromise one makes when employing a concentration technique such as the one used in this paper. In actual casework it would be possible to lose ones valuable sample which is why adhesive tape seems a more reliable method).

The data indicates that large amounts of primer residue may be down range many feet from the muzzle and also many feet to the sides. This observation could become very important in cases where there are several people in a room when a firearm is discharged and one individual is shot. There could possibly be Gunshot Residue on all the individuals while only one individual discharged the firearm. Even with an outside shooting this may come into play. Two cases to illustrate this point can be cited. In one instance, an individual was shot across the hood of an automobile and both persons had Gunshot Residue on them. Also, one elderly woman was shot by her grandson while lying on the couch. The grandson was about 10 feet away. Gunshot Residue was identified on the grandson and grandmother. So, it is important to note that the presence of Gunshot Residue on an individual is not necessarily linked to that individual discharging a firearm but may only indicate physical proximity to a discharged firearm.

From the experimental data there seems to be no connection between an abundance of large

percentages of spheroidal lead particles and being a shooting victim. Several guns used in the data collection were discharged and then the shooters hands were sampled immediately. The percentage of lead particles was substantially the same on the hands as the downrange firings. This does not always coincide with casework where the shooting victims sometimes have high numbers of round spheroidal leads with little or no Pb-Ba-Sb, Pb-Ba or Pb-Sb particles.

However, the ratios of Pb:Pb-Sb:Pb-Ba:unique particles is stable for a particular caliber of firearm. (One might notice the conspicuous absence of any Ba-Sb particles. Sb and Ba have little or no solubilities in each other. Plus, there is usually much more Pb in primers which decreases the probability of having particles without Pb. No Sb-Ba particles were found in all the data). As one might expect, distance does not significantly alter the ratios. It would be interesting to see a study on 2 or 3 different calibers of handguns with many different kinds of ammunition to see how close the different manufacturers may be.

Certain types of firearms do produce higher percentages of unique particles. Also, certain firearms will produce more particles associated with Gunshot Residue. A good example of the latter is the shotgun.

It appears that any attempt to estimate distances from the particle density of a sample would generally not be possible, especially since the amount of Gunshot Residue on an individual declines with time. (If the collection was within a few minutes and the firearm and unfired ammunition were recovered it could be possible to estimate the distance from the victims sample). This would leave the distance estimation always on the long side. The use of particle size would not yield any useful information on distance either. Table V shows the particle sizes of the Gunshot Residue at various distances downrange from the muzzle for the 45 handgun and the 30-06 rifle. There is no significant difference in the particle size of the samples from 5 feet to 14 feet for the 45 and from 8 feet to 17 feet for the 30-06.

As with many Criminalistics (Trace evidence) disciplines, the interpretation of Gunshot Residue on an individual is not always straight forward. The interpretation of the data by the analyst must be given fastidious consideration. Also, the analyst must be very precise and specific in his report writing and subsequent explanation of his findings so that the lay person will not misconstrue the evidentiary implications. Therefore, the awareness of Gunshot Residue deposition in an area surrounding the firearm along with the knowledge that Pb will most always be the largest contributor to Gunshot Residue deposition is crucial to accurate, responsible report writing in shooting cases.

REFERENCES

[1] Sugarman, L., “The Concentration and Isolation of Gunshot Residues for Particle Analysis”, Report B57, American Academy of Forensic Sciences 39th Annual Meeting, San Diego, California, February 1987.

TABLE V

SIZE OF UNIQUE GSR PARTICLES (IN MICRONS)

CALIBER	DISTANCE IN FEET	AVERAGE Particle SIZE (IN U)	NUMBER OF PARTICLES
30-6	8	11.0	5
	11	7.5	2
	14	9.0	4
	17	7.5	2
45	5	9.3	9
	8	10.6	9
	11	12.8	10
	14	11.3	7

TABLE I

CALIBER	DISTANCE (FEET)	Pb	Ba-Pb	Sb-Pb	Sb-Ba-Pb	OTHER	PARTICLE mm2
45	5	69.0%	13.8%	1.7%	15.5%	0.0%	23
45	8	58.4%	7.7%	1.5%	13.8%	0.0%	21
45	11	57.9%	7.9%	7.9%	26.3%	0.0%	56
45	14	59.7%	4.5%	6.0%	11.9%	0.0%	23
30-06	8	78.5%	1.5%	13.8%	6.2%	0.0%	51
30-06	11	89.6%	0.0%	6.2%	4.2%	0.0%	61
30-06	14	78.8%	2.5%	13.8%	5.0%	0.0%	69
30-06	17	86.9%	0.0%	8.2%	4.9%	0.0%	44
SHOTGUN	8	87.5%	8.8%	1.2%	2.5%	0.0%	184
SHOTGUN	11	79.7%	0.0%	8.6%	6.2%	5.5%	15
SHOTGUN	14	85.0%	0.0%	10.0%	5.0%	0.0%	145
38	8	91.4%	1.4%	1.4%	5.7%	0.0%	28
38	11						
38	14	85.0%	0.0%	7.5%	2.5%	5.0%	15
38	17	83.0%	0.0%	13.6%	3.4%	0.0%	28
38	20	85.9%	6.2%	6.2%	1.6%	0.0%	51
22	5	84.0%	5.0%	6.0%	5.0%	0.0%	24
22	8	65.9%	9.1%	11.4%	13.6%	0.0%	10
22	11	66.7%	5.5%	5.5%	22.2%	0.0%	19
22	14	64.3%	7.1%	7.1%	21.4%	0.0%	13
25	5	91.1%	5.9%	2.0%	1.0%	0.0%	26
25	8	80.3%	1.6%	9.8%	8.2%	0.0%	13
25	11	20.7%	6.9%	6.9%	51.7%	13.8%	11
25	14	65.0%	5.0%	5.0%	7.5%	12.5%	6
OUTSIDE RANGE						OUTSIDE RANGE	
38	8	76.8%	2.9%	4.3%	13.0%	2.9%	
38	11	74.3%	0.0%	0.0%	7.7%	17.9%	
38	14	65.0%	0.0%	2.8%	0.0%	7.5%	
38	6'RIGHT	64.0%	4.0%	8.0%	24.0%	0.0%	
38	6'BACK	25.0%	0.0%	0.0%	5.0%	70.0%	
38	6'LEFT	64.3%	1.8%	12.5%	21.4%		
22	11	82.1%	7.1%	3.6%	7.1%	0.0%	18
22	14	87.2%	2.3%	5.8%	4.6%	0.0%	3
30-06	8	17.7%	13.3%	17.7%	24.4%	26.6%	8
30-06	11	28.6%	5.7%	14.3%	0.0%	51.4%	2
30-06	14	47.9%	0.0%	6.2%	4.2%	41.7%	2
INSIDE RANGE						INSIDE RANGE	
38	4'RIGHT	53.0%	7.6%	12.1%	13.6%	13.6%	
38	4'LEFT	79.8%	1.1%	11.2%	3.4%	4.5%	
38	4'BACK	81.0%	2.0%	11.1%	5.0%	0.0%	
38	6'RIGHT	86.0%	1.0%	10.7%	2.3%	0.0%	37
38	6'LEFT	79.6%	0.0%	7.4%	1.6%	11.1%	5
38	6'BACK	90.3%	1.6%	6.4%	1.6%	0.0%	16
38	8'RIGHT	87.8%	3.7%	5.6%	2.8%	0.0%	27
38	8'LEFT	98.9%	0.0%	0.0%	0.0%	1.0%	68
38	8'BACK	89.2%	1.4%	4.3%	2.2%	2.9%	24
	MEAN	71.9%	3.6%	7.1%	9.1%	6.9%	34
	STD DEV	19.5%	3.6%	4.3%	9.8%	14.8%	38
	VARIANCE	3.8%	0.1%	0.2%	1.0%	2.2%	1436

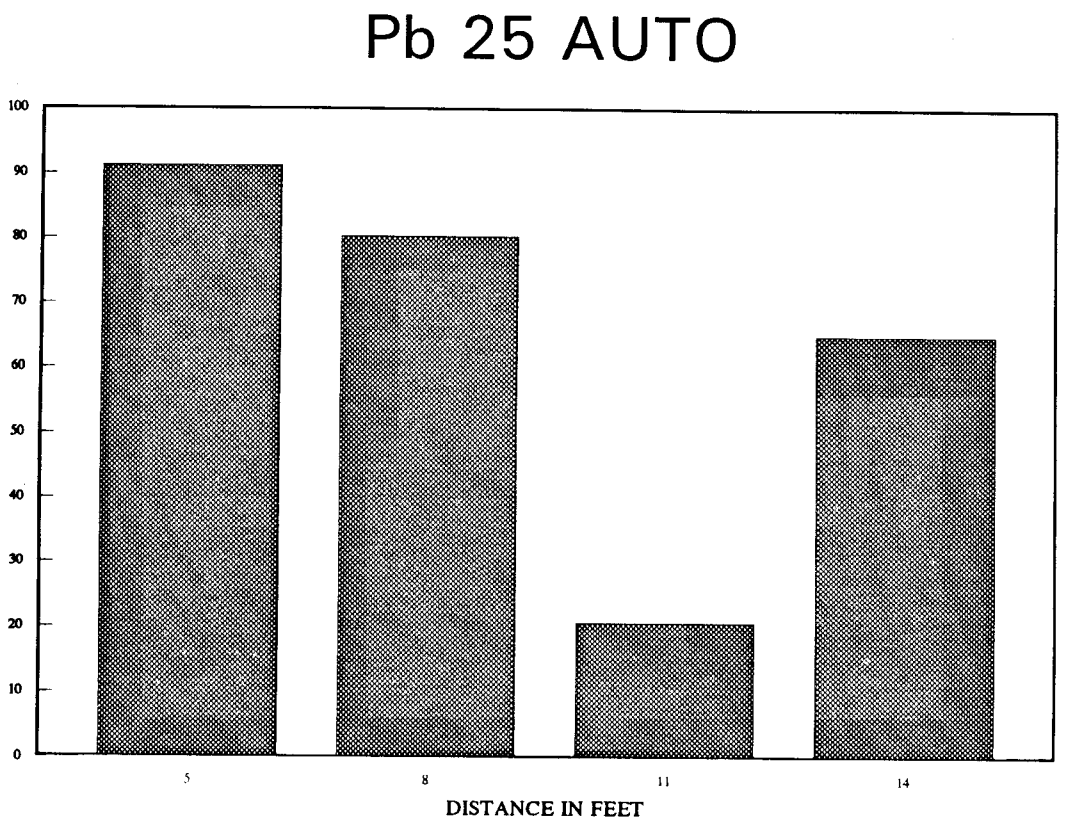
CALIBER	DISTANCE (FEET)	Pb	Ba-Pb	Sb-Pb	Sb-Ba-Pb	OTHER	PAR
		INSIDE RANGE			INSIDE RANGE		
45	5	69.0%	13.8%	1.7%	15.5%	0.0%	
45	8	58.4%	7.7%	1.5%	13.8%	0.0%	
45	11	57.9%	7.9%	7.9%	26.3%	0.0%	
45	14	59.7%	4.5%	6.0%	11.9%	0.0%	
30-06	8	78.5%	1.5%	13.8%	6.2%	0.0%	
30-06	11	89.6%	0.0%	6.2%	4.2%	0.0%	
30-06	14	78.8%	2.5%	13.8%	5.0%	0.0%	
30-06	17	86.9%	0.0%	8.2%	4.9%	0.0%	
SHOTGUN	8	87.5%	8.8%	1.2%	2.5%	0.0%	
SHOTGUN	11	79.7%	0.0%	8.6%	6.2%	5.5%	
SHOTGUN	14	85.0%	0.0%	10.0%	5.0%	0.0%	
38	8	91.4%	1.4%	1.4%	5.7%	0.0%	
38	11						
38	14	85.0%	0.0%	7.5%	2.5%	5.0%	
38	17	83.0%	0.0%	13.6%	3.4%	0.0%	
38	20	85.9%	6.2%	6.2%	1.6%	0.0%	
22	5	84.0%	5.0%	6.0%	5.0%	0.0%	
22	8	65.9%	9.1%	11.4%	13.6%	0.0%	
22	11	66.7%	5.5%	5.5%	22.2%	0.0%	
22	14	64.3%	7.1%	7.1%	21.4%	0.0%	
25	5	91.1%	5.9%	2.0%	1.0%	0.0%	
25	8	80.3%	1.6%	9.8%	8.2%	0.0%	
25	11	20.7%	6.9%	6.9%	51.7%	13.8%	
25	14	65.0%	5.0%	5.0%	7.5%	12.5%	
MEAN		74.5%	4.4%	7.0%	10.7%	1.6%	
STD DEV		15.8%	3.7%	3.8%	11.1%	3.9%	
VARIANCE		2.5%	0.1%	0.1%	1.2%	0.1%	

TABLE III

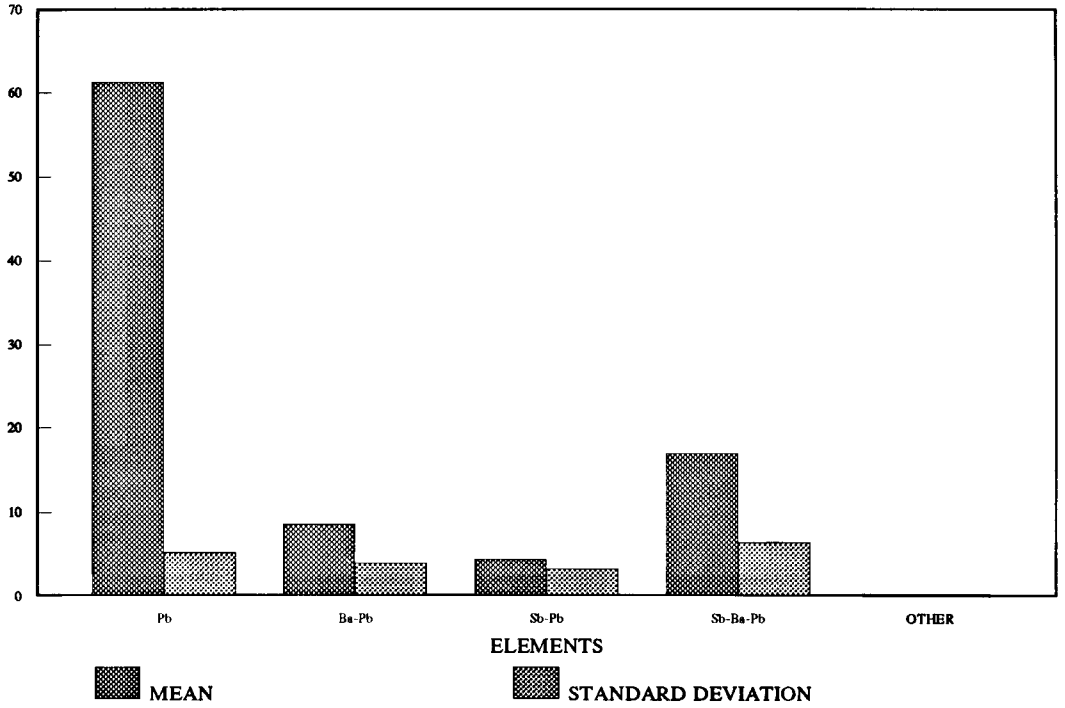
CALIBER	DISTANCE (FEET)	Pb	Ba-Pb	Sb-Pb	Sb-Ba-Pb	OTHER	PAR
		OUTSIDE RANGE			OUTSIDE RANGE		
38	8	76.8%	2.9%	4.3%	13.0%	2.9%	
38	11	74.3%	0.0%	0.0%	7.7%	17.9%	
38	14	65.0%	0.0%	2.8%	0.0%	7.5%	
38	6°RIGHT	64.0%	4.0%	8.0%	24.0%	0.0%	
38	6°BACK	25.0%	0.0%	0.0%	5.0%	70.0%	
38	6°LEFT	64.3%	1.8%	12.5%	21.4%		
22							
22	11	82.1%	7.1%	3.6%	7.1%	0.0%	
22	14	87.2%	2.3%	5.8%	4.6%	0.0%	
30-06	8	17.7%	13.3%	17.7%	24.4%	26.6%	
30-06	11	28.6%	5.7%	14.3%	0.0%	51.4%	
30-06	14	47.9%	0.0%	6.2%	4.2%	41.7%	
MEAN		57.5%	3.4%	6.8%	10.1%	21.8%	
STD DEV		23.1%	3.9%	5.5%	8.8%	23.7%	
VARIANCE		5.3%	0.2%	0.3%	0.8%	5.6%	

TABLE IV

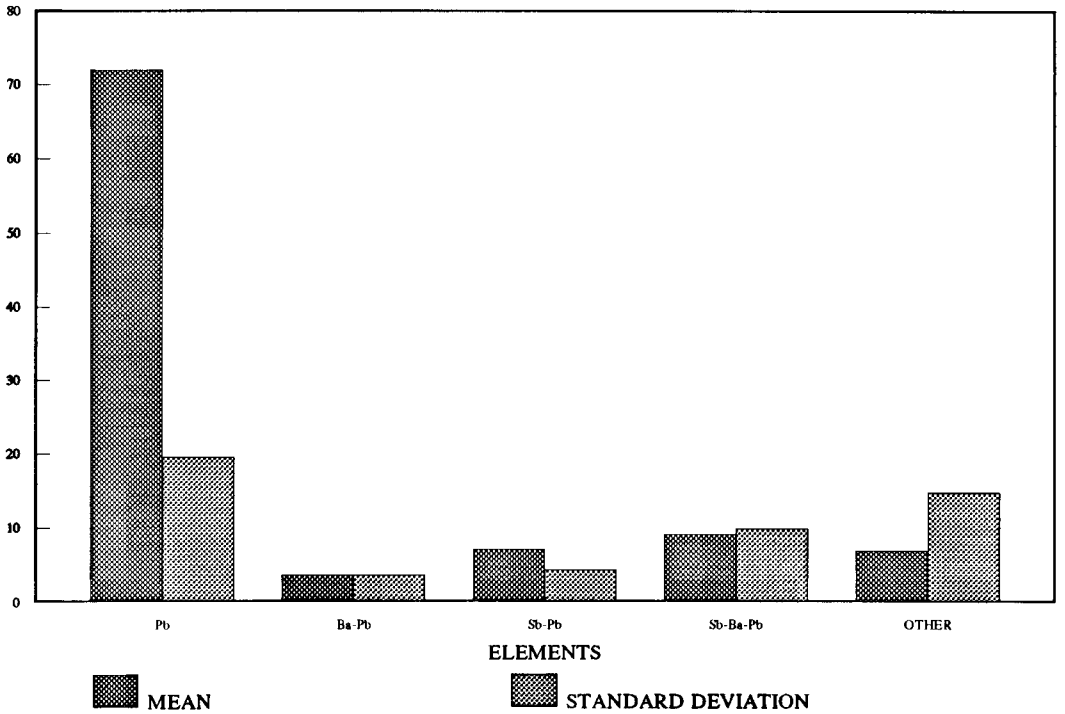
CALIBER	DISTANCE (FEET)	INSIDE RANGE Pb	Ba-Pb	Sb-Pb	Sb-Ba-Pb	INSIDE RANGE OTHER	PARTICLE mm2
38	4°RIGHT	53.0%	7.6%	12.1%	13.6%	13.6%	
38	4°LEFT	79.8%	1.1%	11.2%	3.4%	4.5%	
38	4°BACK	81.0%	2.0%	11.1%	5.0%	0.0%	
38	6°RIGHT	86.0%	1.0%	10.7%	2.2%	0.0%	37
38	6°LEFT	79.6%	0.0%	7.4%	1.8%	11.1%	5
38	6°BACK	90.3%	1.6%	6.4%	1.6%	0.0%	16
38	8°RIGHT	87.8%	3.7%	5.6%	2.8%	0.0%	27
38	8°LEFT	98.9%	0.0%	0.0%	0.0%	1.0%	68
38	8°BACK	89.2%	1.4%	4.3%	2.2%	2.9%	24
MEAN		82.8%	2.0%	7.6%	3.6%	3.7%	29
STD DEV		12.0%	2.2%	3.8%	3.8%	4.9%	20
VARIANCE		1.4%	0.0%	0.1%	0.1%	0.2%	396

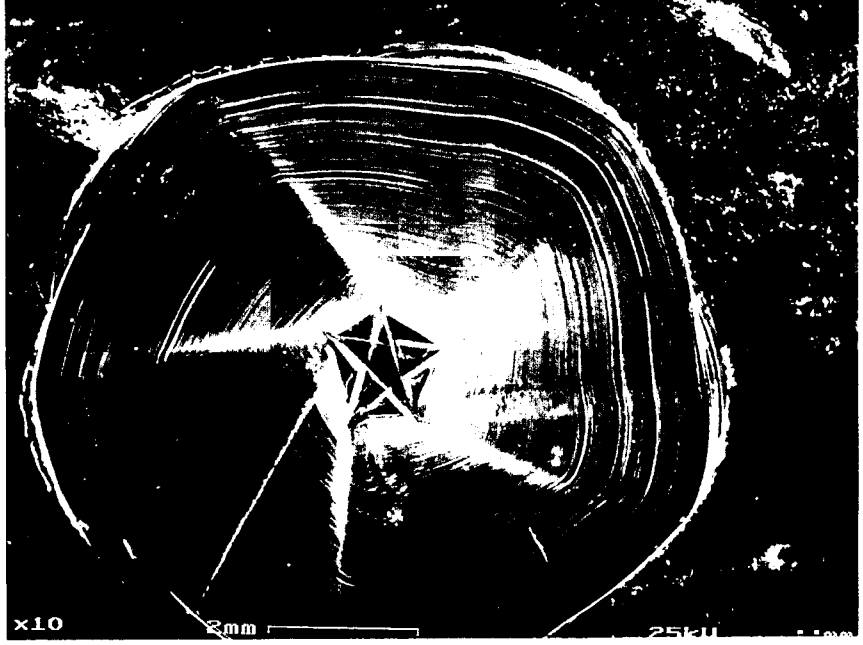


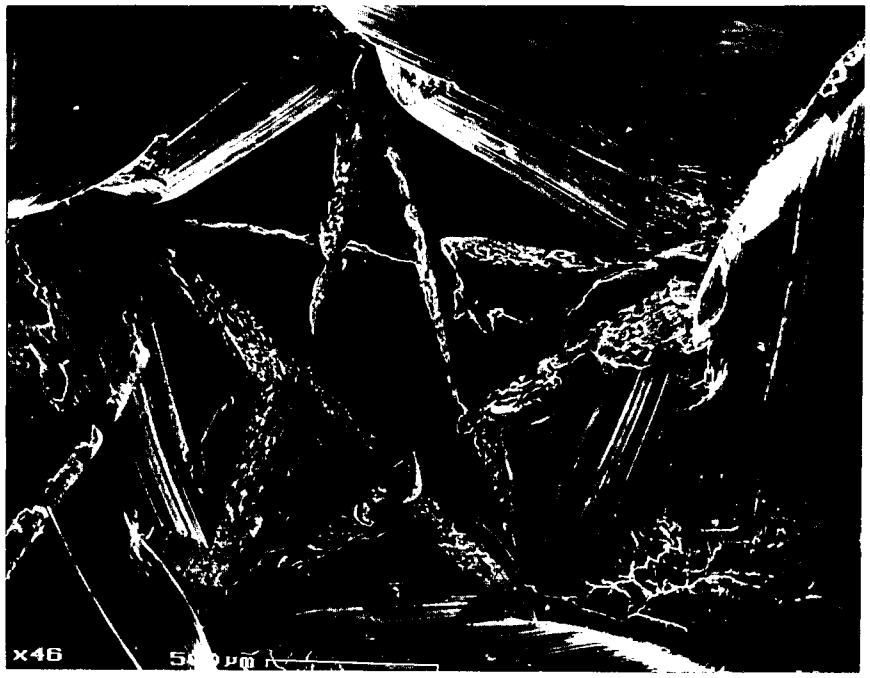
45 S&W 645 INSIDE



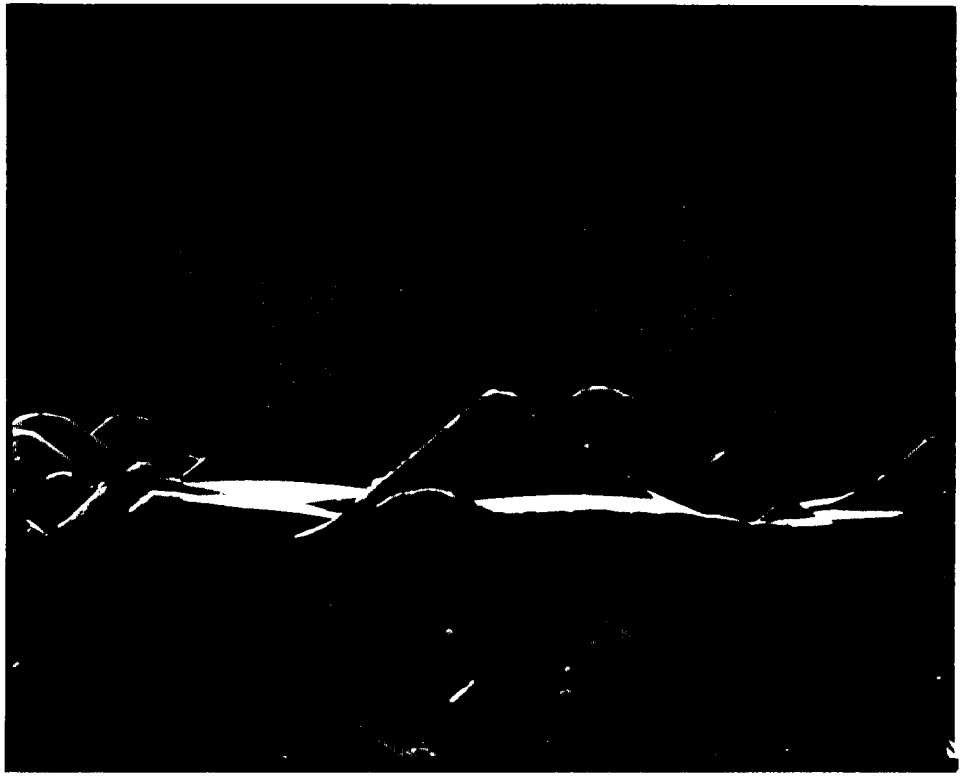
DOWNRANGE INSIDE







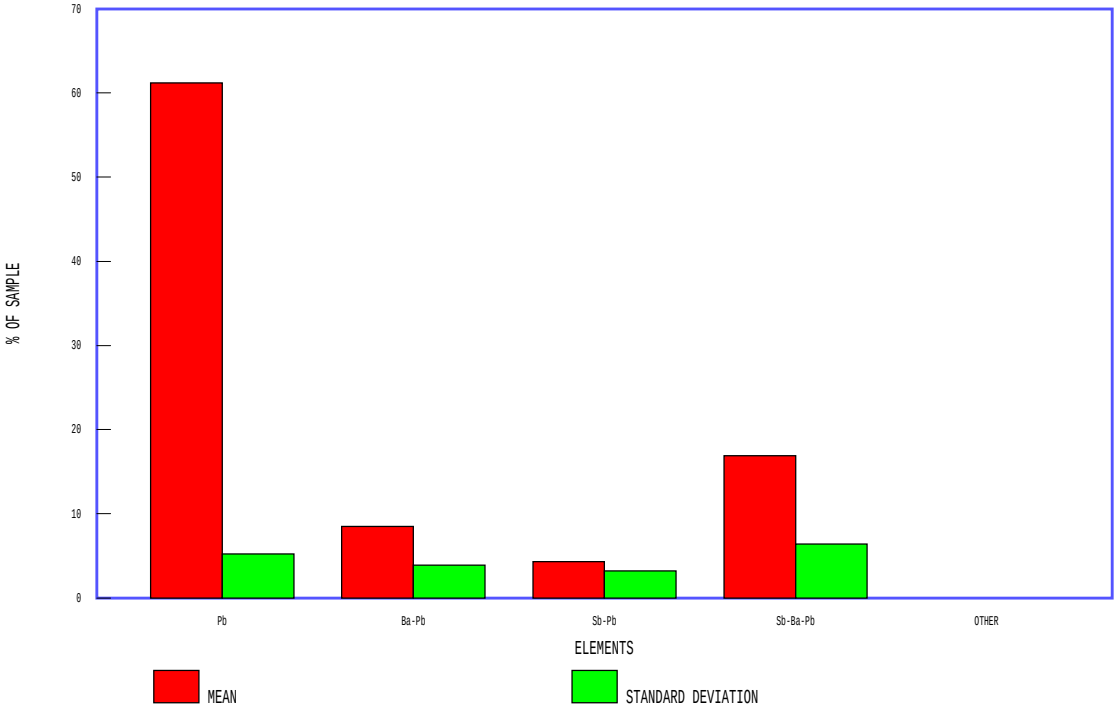
These photos represent an unusual pattern caused by a drill bit tip skipping while boring through aluminum bar stock. The typical pattern normally observed at the bottom of the boring should be triangular. Zeiss 962-DSM 25KV. **Robert Adamo,** Westchester County Forensic Science Lab, NY.



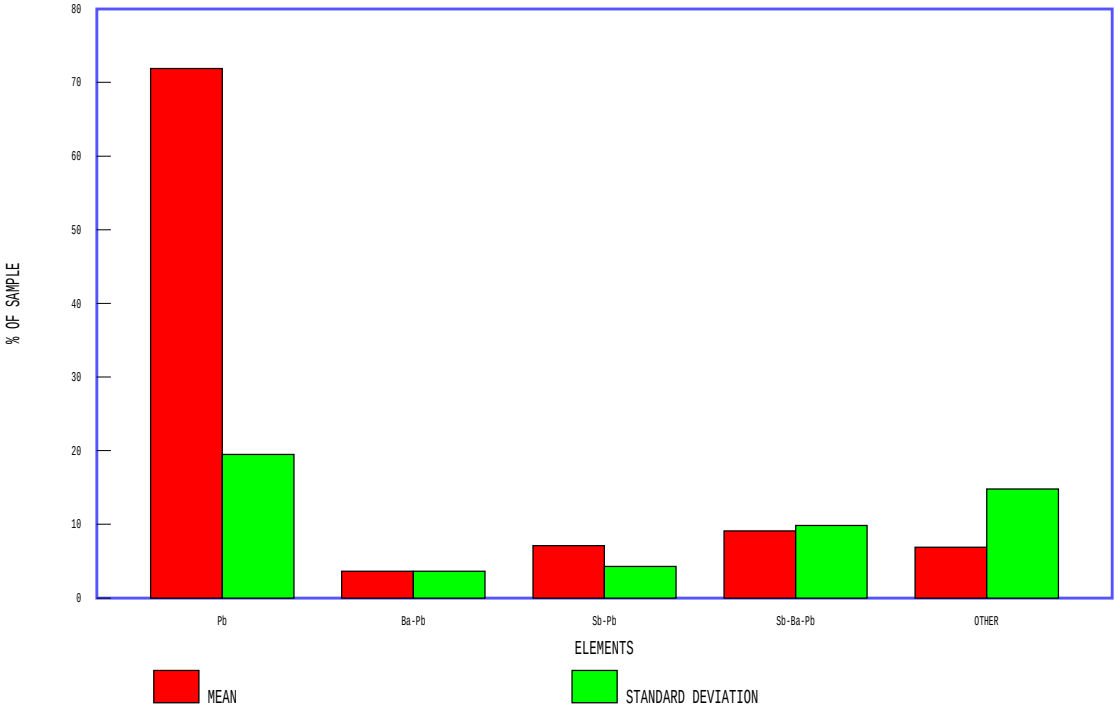
Pink Garnet

...contributed by **Thomas Hopen,**
MVA Inc, Norcross, GA

45 S&W 645 INSIDE



DOWNRANGE INSIDE



WORTH READING

GSR

Tokarev Pistol and Its Cartridge of Chinese Make, Y. Shiotsu, H. Akamatsu, *AFTE Journal* (Vol 26, No 1), Jan 1994.

The cartridge from this pistol has "11 90" headstamp, manufactured in China in 1990. SEM/EDXA of the fired cartridge residue indicates K, Cl, Sb, S, and Sn. Hg was detected by TRXF.

Identification of Ammunitions Used in a Lethal Robbery. Comparison Between Scanning Electron Microscopy/Energy Dispersive X-Ray Analysis (SEM/EDX) and Instrumental Neutron Activation Analysis (INAA) Measurements, A. Moauro and G Falso, *J of Forensic Sciences,* Vol 38, No 5, Sept 1993, p 1237-1242.

The inorganic composition of GSR was used to demonstrate the weapon that fired a fatal shot, with major differences demonstrated by SEM/EDXA and trace diffferences by NAA. Two ammunitions were present, Sellior & Bellot (S&B) containing Pb, Ba, and Sn, and G. Fiocchi (GF)containing Pb, Ba, and Sb. Additionally, muzzle target distance determination was assessed by estimating particle deposition as a function of distance.

Collection Efficiency of Gunshot Residue (GSR) Particles from Hair and Hands Using Double-Sided Adhesive Tape, A. Zeichner and N. Levin, *J of Forensic Sciences,* Vol 38, No 3, May 1993, p 571-584.

Zeichner and Levin compare dabbing to the swabbing/comb method for GSR collection from hair and find no differences in collection efficiency. Maximum collection efficiency is achieved after 200-300 dabbings although stickiness appears to expire after 100 dabbings. 50 to 100 dabbings are necessary to achieve the maximum collection efficiency from hands although stickiness appears to expire after 20 to 30 dabbings. They have found GSR in hair samples when GSR was not found on hand samples.

EDX

X-ray Continuum Discrimination Technique for the Energy Dispersive Analysis of Fine Particles, LE Giblin et al, *Analytical Chemistry*, Vol 65, No 24, Dec 15, 1993.

Because background continuum varies with orientation at the point of analysis, irregularly shaped grains can be selected for quantitative EDX based on a simple ratio of high-low energy background windows.

Sleuthing the Impossible Spectrum, Z. Corder, *Spectroscopy* 9(1), Jan 1994, p 33-35.

An "unusual spectrum" with minimal counts in the low energy range was found to be caused by a part of the sample blocking the path to the detector (resulting in absorption of low Z x-rays). We wouldn't do this - would we?

Cathodoluminescence

Applications of Real Color Cathodoluminescence Observations in the Scanning Electron Microscope, G Saparin and S. Obyden, *USA Microscopy and Analysis*, January, 1994, p 7-9.

Developments in CL interfaced with the SEM (CCL-SEM) permit "real color" contrast and fast (6 sec) scans, integrating morphological and CL spectral information in real time. Several applications (primarily in semiconductor technology) are listed.

Archeology

Biodeterioration of Ancient Textiles: SEM Characterization, M Romano, et al, *USA Microscopy and Analysis*, January, 1994, p 19-21.

The SEM is used to characterize fiber degradation of historic textiles during the restoration steps of cleaning and preserving. Several methods for cleaning and preservation are presented. References appear useful.

Ink

Application of the Microdroplet Method of X-ray Analysis to the Characterization of Inks, M. Cassidt and D. Allen, *J of Forensic Sciences,* Vol 38, No 1, Jan 1993, p 40-47.

Analysis of 17 ballpoint pen inks extracted by pyridine/ethylene glycol and deposited on organic film showed no detectable elements by SEM/EDX. Discrimination between four fountain pen inks was possible.

MEETINGS, SCHOOLS . . .

(April 1994)

Advanced Materials. Apr 11-14 (Lake Bluff, IL), Nov 7-10 (Tucson, AZ). Institute for Microstructural Analysis, Buehler Ltd, Lake Bluff, IL. Preparation and characterization techniques for microscopic analysis, including SEM. 708/295-4659.

Southern Association of Forensic Scientists (SAFS) and Southwestern Association of Forensic Scientists (SWAFS), April 12-16, 1994, Little Rock, AR. *Workshop on SEM/EDX*. Ken Michau or Gary Dallas, 501/2275747.

May 1994

Mid-Atlantic Association of Forensic Scientists (MAAFS), May 4-6, l994, Virginia Beach, VA. Harry Finley or Marc Jaskolka, NCIS, 9079 Hampton Blvd, Norfolk, VA 23505.

Scanning Microscopy 1994, May 7-12,1994, Toronto (Downtown, City Hall) Canada. Contact Dr. Om Johari, Scanning Microscopy International, 708/529-6677.

California Association of Criminalists (CAC), Spring Seminar, May 11-14, 1994, Oakland, CA. Contact Mary Gibbons, Oakland PD Lab, 510/238-3386.

SCANNING 94 joint meeting with SEEMS 94, May 17-20,1994, Charleston, SC. Symposium "*Applications of Scanning Microscopy in Forensic Sciences"*. Contact Mary Sullivan, 201/818-1010.

June 1994

Lehigh Microscopy Short Courses, June 13-23, 1994, Bethlehem, PA. Contact David Williams, 215/758-5133. SEM & X-ray microanalysis.

ICDD (International Centre for Diffraction Data) Clinic on X-ray Fluorescence Spectrometry, *Fundamentals of XRF,* June 20-24, *Advanced Methods in XRF*, June 27-July 1, Newtown Square, PA. 610/325-9814.

July 1994

INTER/MICRO-94, July 18-21, 1994, McCrone Research Institute, Chicago, IL, 312/842-7100. Contact Nancy Daerr to contribute a 10-15 min original paper.

MAS/MSA 1994, July 31-August 5, 1994, New Orleans, LA. Contact: MSA Meeting Office, 800/538-3672.

August 1994

Denver X-ray Conference, August 1-5, 1994, Steamboat Springs, CO. Lynne Bonno, Dept of Engineering, U of Denver, Denver, CO 80208.

September 1994

Micro94, Sept 12-15, 1994, London, England. Royal Microscopical Society, Tel: (0865) 248768

October 1994

SEM and X-Ray Microanalysis for Materials Science: An Introductory Course, Oct 10-14, 1994. State University of New York at New Paltz. 914/257-3800.

Midwestern Association of Forensic Scientists (MAFS), Oct 11-16, 1994, Cleveland, OH. Mary Wenderoth or Cathy Denissoff, 216/623-5646.

California Association of Criminalists (CAC), Fall Seminar, Oct 19-22, 1994, Pasadena, CA. Contact Manuel Munoz or Dan Anderson, Los Angeles County Coroner, 213/343-0530. (This will be the first joint CAC/Forensic Science Society meeting!)

Canadian Society of Forensic Science (CSFS) and Northwest Association of Forensic Scientists (NWAFS), October 31-November 5, 1994, Vancouver, British Columbia, Canada. Jeffrey Coughlin, 604/264-3507.

November 1994

Advanced Materials, Nov 7-10, 1994, Buehler Ltd. Tuscon AZ. 708/295-4659. See listing under April.

Australia and New Zealand Forensic Science Society, November 21-25, 1994, Auckland, New Zealand. Douglas Elliot, Auckland, New Zealand, 09-815-3670.

Southwestern Association of Forensic Scientists (SWAFS) will hold its Fall 1994 training seminar at the Adam's Mark Hotel in Houston, TX, Nov 15-19, 1994. A variety of workshops, *including SEM/EDX.* Pauline Louie, Houston Police, 713/247-5449

MAS 1995, Denver, CO
MSA 1995 August 13-18 Kansas City, KS
MAS/MSA 1996, Minneapolis

. . . And this from the Carl Zeiss newsletter "TOPICS", issue No. 4:

Stability pays off:

Scanning electron microscopes from Carl Zeiss become standard equipment in forensic laboratories

In recent years automatic gunshot residue analysis has seen widespread acceptance by the forensic laboratories. This technique allows quantitative detection of the distribution and chemical composition of any residual particles deposited on the hand of an individual who has fired a gun. As the distribution and composition of the gunshot residue pattern is different for every weapon, the technique can be used for identification purposes. The first step involves the transfer of all particles in a 1:1 contact process from the palm on the hand to a sticky tape. This is then inserted in a scanning electron microscope for automatic inspection. For this purpose, the SEM is placed under the direct control of an energy-dispersive X-ray spectrometer equipped with dedicated software. A map is then generated showing the exact local distribution and chemical composition of all detected gunshot residue particles on a surface of typically (10X10) cm2.

The small size of the particles to be detected and the high measurement accuracy required means that the generation of such a map takes many hours, thus putting very high demands on the long-term stability and the reliability of the SEM. Under these extreme conditions, the outstandingly high standard of Carl Zeiss's technology in electron optics, digital control, precision stage mechanics and turbomolecular pumping proves to be an invaluable asset. Impressive image comparison capabilities of the Zeiss-SEMs - and especially the DSM 962 - by means of their integrated frame store complete the picture.

As a result, more than two thirds of all forensic laboratories in Germany have opted for a DSM from Carl Zeiss: an impressive confirmation of our never-ending quest for high quality.

...contributed by Jon Kokanovich, Mesa Police Dept, Mesa, AZ

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