

*V. R. Matricardi,<sup>1</sup> Ph.D. and J. W. Kilty,<sup>1</sup> B.S.*

## Detection of Gunshot Residue Particles from the Hands of a Shooter

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The identification of gunshot residue (GSR) is a topic of great interest to the police officer and prosecutor and a problem for the criminalist. The police officer and prosecutor want a test that will demonstrate the existence of GSR on a subject's hands. The difficulty for the criminalist is in associating the quantitative amounts of the GSR-indicating elements (barium, antimony, and lead) exclusively with GSR. In this paper a technique is described that tries to identify as GSR the individual particles which are part of the components of the smoke emanating from the discharge of a firearm.

The method consists of collecting particles from the shooter's hand and individually examining some of the particles with the scanning electron microscope and its associated X-ray analyzer for elemental composition and characteristic appearance. If proper studies from the hands of persons who have not fired a weapon ("handblanks") prove the uniqueness of these particles, the criminalist will be able to state unequivocally that GSR is present. In addition, the technique is essentially nondestructive, thereby allowing for subsequent quantitative analyses.

It should be stated at the outset that the presence of GSR on an individual's hand does not necessarily prove that the person discharged a firearm. The person could have been in close proximity to the discharge, or, as will be shown later, may have entered the room after the discharge.

In 1971 E. Boehm [1] presented micrographs of GSR particles on fabric located close to the muzzle. Boehm stated that both the analysis and morphology of these particles were important for identification and that the morphology at higher magnifications would differ depending on the ammunition used. Nesbitt et al [2,3] have provided a thorough study of particulate gunshot residue found on the hand of the shooter using the "tape lift" technique. Diederichs et al [4] provided a short study of the particulate residue from various .22-caliber rimfire cartridges picked up from the shooter by a cotton swab. Another more recent and thorough study has also been published by Andrasko and Maehly [5].

Because of the encouraging initial results and the interest thereby generated, a symposium on particulate GSR was held at Aerospace Corporation [6] under the auspices of the Law Enforcement Assistance Administration. Attending were 19 individuals associated with crime laboratories who had an interest in the subject. One of the authors (V. R. M.) attended this symposium to determine the status of the research and the prevalent experimental methods. The decision had already been made by the authors that the technique showed promise and that an in-house evaluation and a correlation between the number of particles and the quantitative results obtained by neutron activation analysis (NAA) were to be made.

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<sup>1</sup>Research physicist and special agent, respectively, Elemental Analysis Unit, FBI Laboratory, Washington, D.C. 20535.

The techniques currently used at the authors' laboratory to determine if a person has been exposed to the discharge of a firearm consist of swabbing the back of the thumb, forefinger, and connecting web area of each hand [7] and determining the amount of antimony and barium by using NAA. The conclusions reached are based on the amounts of antimony and barium as compared to published and unpublished handblank values, the quantitative difference in antimony and barium between the right and left hands, the amount of time elapsed between the alleged discharge and the swabbing, and the amount of deposit expected from a given gun.

What is presented here is an interim report of a technique that is still being developed. Included are various attempts we took but did not adopt; we state them to save others similar effort. Some of this work has already been presented elsewhere [8,9]. A description of the morphology of GSR particles has been left out since the appearance of the larger particles has been thoroughly described in the literature [2]. In addition, a correlation to existing NAA data is found and allows a prediction to be made of the number of particles expected from the discharge of a firearm.

## Materials and Methods

### *Firing of Weapons*

The weapons were test-fired into a bullet recovery box at the FBI laboratory. All of the firings were performed in the morning to reduce the possibility of residual airborne contaminations. The muzzle of the weapon was held at least 0.5 m away from the cardboard face of the bullet recovery box. The weapons used in this study were these:

- (1) a .45 Auto U.S. pistol, Model 1911A1, 5 in. (127-mm) barrel, #E17 (same weapon as used in previous study);
- (2) a .38 Special Charter Arms revolver, Model Undercover, 2-in. (51-mm) barrel, #D563 (same chamber used in all firings);
- (3) a .25 Auto Colt pistol, Model Junior Colt, 2-in. (51-mm) barrel, #B22; and
- (4) a .22 caliber Harrington and Richardson revolver, Model 622, 2½-in. (64-mm) barrel, #A237 (same chamber used in all firings).

The ammunition used in the study is listed in Table 1. In most cases the weapons were

TABLE 1—*Ammunition used in the study.*

Caliber	Manufacturer	Bullet	Primer
.45 Automatic	Remington	230-grain, full metal case, round nose	Uleah bore
.45 Automatic	Federal	230-grain, full metal case, round nose	nonmercuric, noncorrosive
.38 Special	Winchester	158-grain, lead, round nose	noncorrosive
.38 Special	Remington	158-grain, lead, round nose	Uleah bore
.38 Special	Remington	125-grain, jacketed hollow point	...
.38 Special	Peters	158-grain, full metal case, round nose	...
.25 Automatic	Remington	50-grain, full metal case, round nose	Uleah bore
.25 Automatic	Winchester	50-grain, full metal case, round nose	noncorrosive
.22 Long Rifle	Federal	40-grain, copper coated, lead, round nose	noncorrosive

cleaned prior to firing. A study was also attempted on the effect prior firings have on GSR deposition.

### *Method of Collection*

The collection for NAA was performed with cotton swabs moistened with 5% nitric acid (pH 1.1) by the method described by Kilty [7]. The NAA examination was performed by standard techniques already described.

The collection for the scanning electron microscope (SEM) examination was similar to that previously described [3]. Pieces of aluminum foil [0.001-in. (0.025-mm) gage] attached to a 25-mm diameter aluminum SEM specimen stub were used. A layer of Scotch Brand 465 adhesive was placed on the aluminum foil and used as the collecting surface. The stub was then dabbed repeatedly over the back of the thumb, forefinger, and web area of the hand until the stickiness of the tape was lost.

After the collection the surface of the adhesive was coated with a conductive layer as soon as possible. This serves the double purposes of reducing the probability of surface capture of particles from the atmosphere and of preventing charging effects during SEM examination.

### *Method of Examination*

A preliminary light microscopy examination of the specimen could often localize the larger GSR particles. These consisted of light brown specimens (up to a millimetre in size) and darker spheres from 10 to 50  $\mu\text{m}$  in diameter. These particles could be marked for subsequent SEM examination.

An Etec scanning electron microscope having both wavelength (Etec) and energy dispersive (Kevex 5100) X-ray analyzers was used for these experiments. The acceleration voltage used was 30 kV.

The specimen was inserted and the surface was tilted roughly 30 deg to the horizontal. The specimen was viewed in the "reduced area" mode, which scans at a rate of roughly 10.5 frames/s. This was chosen instead of the television mode suggested [2] because the image is portrayed in the standard viewing screen (not on an auxiliary monitor), reducing the time required to switch between low magnification scanning and high magnification viewing (or analysis). At 10.5 frames/s the specimen can be translated at a comfortable rate for particle recognition.

One of the the first tasks of this project was to determine how easily the GSR particles could be recognized. To enable the researchers to become familiar with the appearance of the particles, a piece of adhesive was placed on the back of the firing hand prior to shooting. In this case the .45 pistol with Remington ammunition was used. Small areas of the tape were dissected and each section was searched to determine the number, size, and analysis of each particle. An average of 3.4 particles/ $\text{mm}^2$  per shot was found on the tape. This figure was improved considerably with practice and with the development of new searching techniques that allowed even smaller particles to be found. Even under these ideal conditions, roughly 25% of the particles found did not contain any of the following elements: lead, barium, antimony, copper, or zinc. Those particles that could not be associated with GSR tended to be composed of silicon, calcium, and iron. The search for GSR particles with elemental mapping to detect high concentrations of lead, barium, or antimony proved unworkable because of the long time required, the low resolution, and the high background (the size of the particle detected by this technique has to be roughly one tenth of the viewed area).

An attempt was also made to determine if the antimony, barium, and lead could be recognized as being on the specimen by nondestructive bulk analysis techniques prior to

searching for the actual particles. The results would have provided an initial criterion by which to decide if the specimen required further examination by particle search. Analysis by both X-ray induced X-ray fluorescence and the SEM-energy dispersive X-ray (EDX) system on the full specimen surface provided inconclusive results and had to be abandoned.

From experience it was found that the information obtained by EDX was superfluous above 12.7 keV. The "fine gain" of the pulse processor control was varied until the lead  $L_{\beta}$  peak was at full scale. This process provided quick recognition of the identity of peaks of interest (lead, barium, antimony, calcium, iron, copper, and zinc).

Various methods of GSR particle recognition were attempted because the normal (secondary electron) image did not distinguish the GSR particles from contaminants and epithelial cells clearly enough.

By using normal secondary imaging (secondary electron image approach) the electron beam current (about  $2 \times 10^{-10}$  A) was varied until a wide field scan of the specimen (about  $\times 30$ ) provided about 800 counts/s in the X-ray energy dispersive spectrometer. With this electron current setting the magnification was increased to  $\times 100$  to  $\times 500$  (in order to recognize the smaller particles) and the specimen was scanned. If a "suspicious" particle were noticed, the magnification was increased until the full electron beam was played on it, and if the counts per second increased appreciably (from 800 to roughly 3000) the particle would be analyzed by EDX. Actually, instead of monitoring the counts per second output, a light-emitting diode (LED) in the preamplifier of the Kevex unit was physically moved to the periphery of the SEM viewing screen. The frequency of the flicker in this LED is proportional to the counts per second. While viewing the particles on the screen, the observer was able to use peripheral vision to note if a particle met the counts per second criterion. However, not all particles having high counts per second are GSR particles, but the other particles may consist of silicon, calcium, or iron. The main disadvantages with this technique are the time and experience factors. Experience is required to detect suspicious particles and time is required to home in and verify with the X-ray analyzer. Thus a new method of recognition of the particles was sought.

An attempt to improve the ability to recognize these particles relied on the alteration of the scan generator to sweep the beam at a very slow scan speed. The counts per second detected by the Kevex energy dispersive X-ray detector was used to indicate when the beam interacted with a suspected GSR particle. The X-ray production goes up with the atomic number  $Z$  of the specimen, and the GSR particles (containing lead, antimony, and barium) generally have a higher average  $Z$  than the background. This technique proved too slow for routine use.

The backscattered electron production is also dependent on the atomic number of the specimen, thereby making it useful for the detection of GSR particles. Figure 1 shows a low magnification image of a piece of tape that has been dabbed on the back of the firer's hand under both secondary (*a*) and backscattered electron imaging (*b*). The bright particles in the backscattered image are, in this case, all GSR particles. Other particles having a high iron or calcium concentration may provide false positives, but subsequent X-ray analysis will eliminate these. With a small amount of experience an operator can routinely detect the GSR particles using the backscattered electron detector. To increase the signal the detector should be located near the incident beam, the distance between the specimen and the detector should be made as small as possible, and the beam current has to be increased above that required for secondary imaging. (In some cases these adjustments may require changing the distance and the aperture to the X-ray detector.)

In this study the backscattered image approach was used to detect GSR particles in the tape lift. The criteria used for initial recognition of these particles were brightness of the backscattered image, sharpness of the edges of the image, sphericity for the smaller particles, and the counts per second seen by the X-ray detector (roughly four times that of background). While only one of these criteria needs be met for an X-ray analysis to be taken

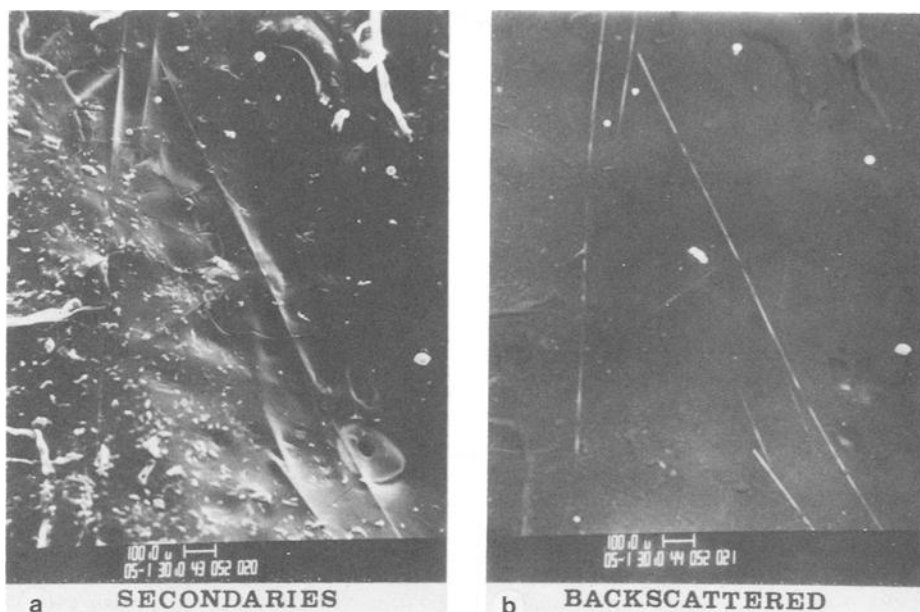


FIG. 1—(a) Tape specimen after dabbing viewed using secondary electron imaging; (b) same area viewed using backscattered electron imaging showing high contrast of particles having a high average atomic number. The bars at the bottom of the micrographs represent 100  $\mu\text{m}$ .

for verification it is still possible that some GSR particles are undetected. Note that at the fast scanning rates the backscattered electron image becomes elongated in the direction of the scan.

## Results

Under optimum conditions, dabbing immediately after firing a dirty gun, it took an average of 1 min to locate and analyze one GSR particle. It should be emphasized that this time requirement will be much longer in actual case conditions where a finite time has elapsed between the firing of a weapon and the collection, thereby reducing the number of particles per unit area of specimen. Kilty [7] found that the amount of barium on the hands of a shooter decreases by a factor of ten in the first 2 h of normal activity (no washing).

For the .45 pistol more than 70% of the particles have characteristic dimensions of less than 5  $\mu\text{m}$ . Figure 2 shows the size distribution of the GSR particles deposited onto a piece of tape placed on the back of the hand prior to firing. From Figure 2 it can be noted that 55% of the particles contain only lead and that most of the particles less than 1  $\mu\text{m}$  in diameter are lead spheres. An item not shown in Fig. 2 is that the percentage of particles in which all three elements (barium, antimony, and lead) are evident is less than 5%. However, this is partially an instrumental limitation. With this ammunition (Remington .45) there is an appreciable amount of calcium present in the residue. When examined with EDX calcium has peaks that overlap the antimony peaks. To emphasize this instrumental limitation Fig. 3 portrays a GSR particle having a calcium peak in its EDX spectrum and no apparent antimony. When the particle is analyzed with the higher resolution wavelength dispersive spectrometer the presence of antimony is evident. This advantage of the wavelength dispersive analyzer still does not, in our opinion, offset the speed advantage of the EDX. However, in the case of a specimen having only a few particles containing barium or

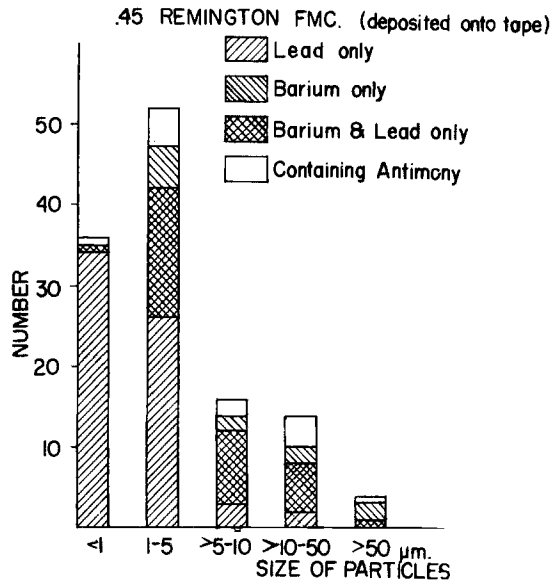


FIG. 2—Size and element distribution of GSR particles deposited onto a piece of tape placed on the back of the firing hand.

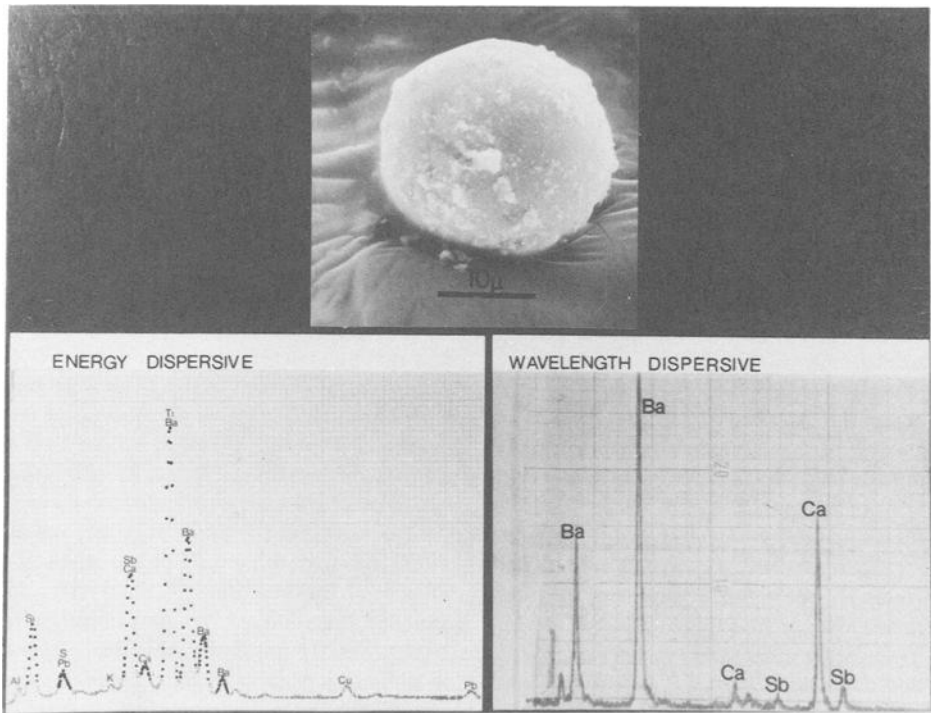


FIG. 3—A GSR particle with associated X-ray spectra obtained with energy dispersive (EDX) and wavelength dispersive (WDS) spectrometers. The spectrum from the WDS is able to resolve the small antimony peaks that are completely buried beneath the calcium peak in the spectrum from EDX.

barium with lead and calcium, reexamination of these particles for antimony with the wavelength spectrometer would provide an increased confidence in the presence of GSR. Similar overlaps occur for titanium and barium as well as for lead and sulfur. This should not be misinterpreted to mean that these elements cannot be distinguished from each other. Usually the peak shapes and other less intense nonoverlapping peaks will eliminate the ambiguity. However a small amount of one element (for example, antimony) can be masked effectively by the presence of a larger or equal amount of another (for example, calcium).

The firing of the various weapons followed by immediate dabbing of the hand produced the results shown in Fig. 4. To be noted again is the high incidence of small lead spheres. The source of the submicrometre lead spheres could be either the lead styphnate in the primer or parts of the bullet that have been melted during the firing. To determine this source a .38 revolver was fired with Remington jacketed hollow point (JHP) ammunition (Fig. 4*h*). This jacketed ammunition does not expose any lead surface to either the barrel or the hot gases of the propellant. The same weapon firing lead bullets provided the most populous particle category in the submicrometre range (Fig. 4*c*). If the JHP ammunition were used the 1 to 5- $\mu$ m diameter nonspherical particles became more populous, with just half of these being lead only. Thus the conclusion can be drawn that the origin of the submicrometre particles is the lead styphnate in the primer and the lead of the bullet. The spherical lead particles seem to originate with the bullet. Is the presence of the lead microspheres caused by melting of the surface of the lead bullet during the explosion or is it due to the heat of friction between the bullet and the barrel? To answer this question, fully clad Peters ammunition was fired with the same gun. This ammunition has no jacket over the base of the bullet and therefore lead is exposed to the explosion. The results from these firings (Fig. 4*i*) were not appreciably different from the results obtained with JHP ammunition in which no lead is exposed to the explosion (Fig. 4*h*). The tentative conclusion is that the thermal effects on the back of the lead bullet do not contribute appreciably to the lead concentration of the GSR particle distribution.

Each one of the tape dabbings obtained during the collection of the data shown in Fig. 4 was analyzed for the quantity of barium and antimony by NAA. The goal was to correlate the quantity of barium to the number of particles of GSR found. The data shown in Table 2 show the results of these comparisons. The next to the last column represents the mass of barium expected from the average GSR particle. This number was obtained by counting the number of barium-containing particles per square millimetre, and from this the expected number on the whole stub was calculated; NAA provided the mass of barium present in the known area of the stub. To determine the amount of barium (in nanograms) per barium-containing particle the amount of barium in  $\text{ng}/\text{mm}^2$  was divided by the number of particles per square millimetre.

There is an appreciable error associated with this type of experiment because of the lack of uniformity in the distribution of the particle deposition. However, the numbers that have been obtained can be used to correlate the extensive NAA data to the expected number of particles. As an example, we know that the .45 pistol with Remington ammunition deposits, on the average, 9.7  $\mu\text{g}$  of barium. From Table 2 we find there are 7 ng of barium for each particle containing barium. Thus 1400 particles containing barium are expected to be found. Note that the standard deviation for the number of particles is 1000.

In a separate experiment several of the larger spheroids, about 30  $\mu\text{m}$  in diameter, were picked off the hands of a shooter (.45 pistol with Remington ammunition) and analyzed by NAA. Figure 5 shows two of the particles, with the results from NAA. The SEM provided the measurements of the volume and NAA provided the quantity of barium and antimony present. Dividing one by the other, the particles contained between 2.7 and 8  $\text{g}/\text{cm}^3$  of barium and the ones containing antimony had between 0.3 and 1  $\text{g}/\text{cm}^3$ . These numbers rely on the assumption that the barium and antimony are uniformly distributed throughout the particles.

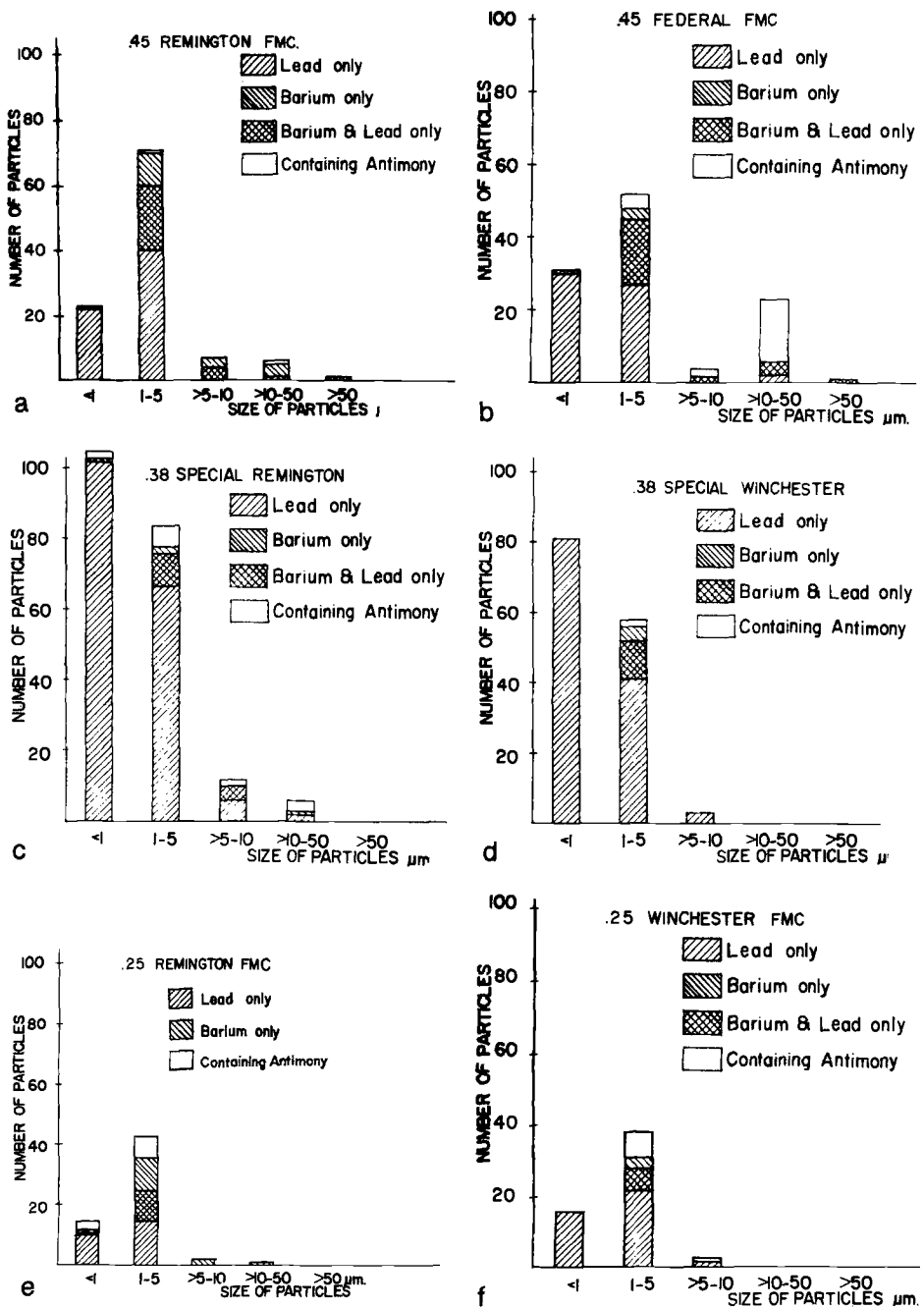


FIG. 4—a through f: Size and element distribution of GSR particles found on dabbings obtained from firing hand using various hand weapons and ammunition.

If the density of barium in the particles and their size distribution are known it is possible to calculate the number of particles from the total quantity of barium deposited, as determined by NAA.

Again this calculation was performed for the .45 pistol with Remington ammunition ( $10 \mu\text{g}$  barium deposited) and the number of particles expected agrees with the 1400 figure that was previously obtained.



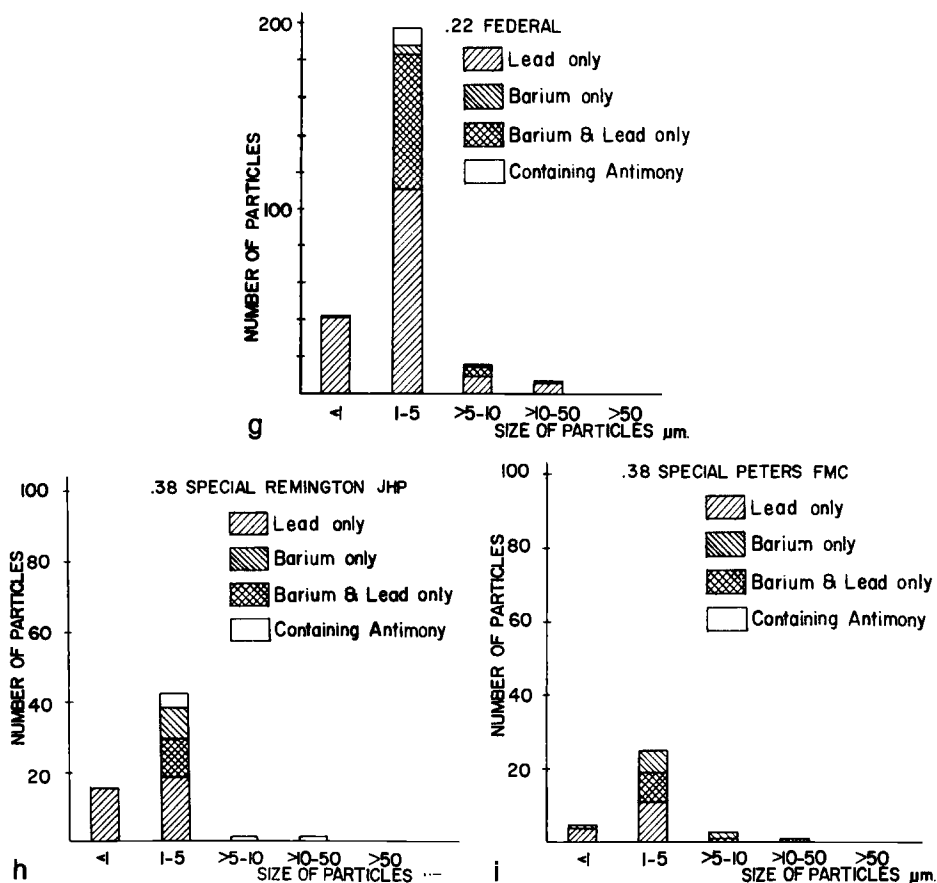


FIG. 4—g through i: Size and element distribution of GSR particles found on dabbings obtained from firing hand using various hand weapons and ammunition.

TABLE 2—Correlations between quantity of barium and number of GSR particles found.<sup>a</sup>

Weapon and Ammunition	Barium-Containing Particles, <i>n</i>	Amount of Barium per Barium-Containing Particle		Standard Deviation
		Range, ng	Average, ng	
.45 pistol Remington FMC	29	0.05–11.0	7	5
.45 pistol Federal FMC	50	0.8–11.0	4	6
.38 revolver Remington	34	0.4–31	10	13
.38 revolver Remington JHP	32	0.25–11	3.5	3.5
.38 revolver Peters FMC	19	1–6	3	3
.38 revolver Winchester	18	0.25–0.7	0.3	0.2
.25 pistol Remington	34	0.4–5	2.4	1.8
.25 pistol Winchester	13	3–27	9.6	10
.22 pistol Federal	119	0.02–1	0.4	0.38

<sup>a</sup>FMC = full metal case; JHP = jacketed hollow point.

The number of GSR particles in the stub (estimated from the number of particles found per square millimetre) varies from 400 to 2500, an agreement (within the experimental error) with the 1400 particles predicted. Thus if deposition of barium in a form other than

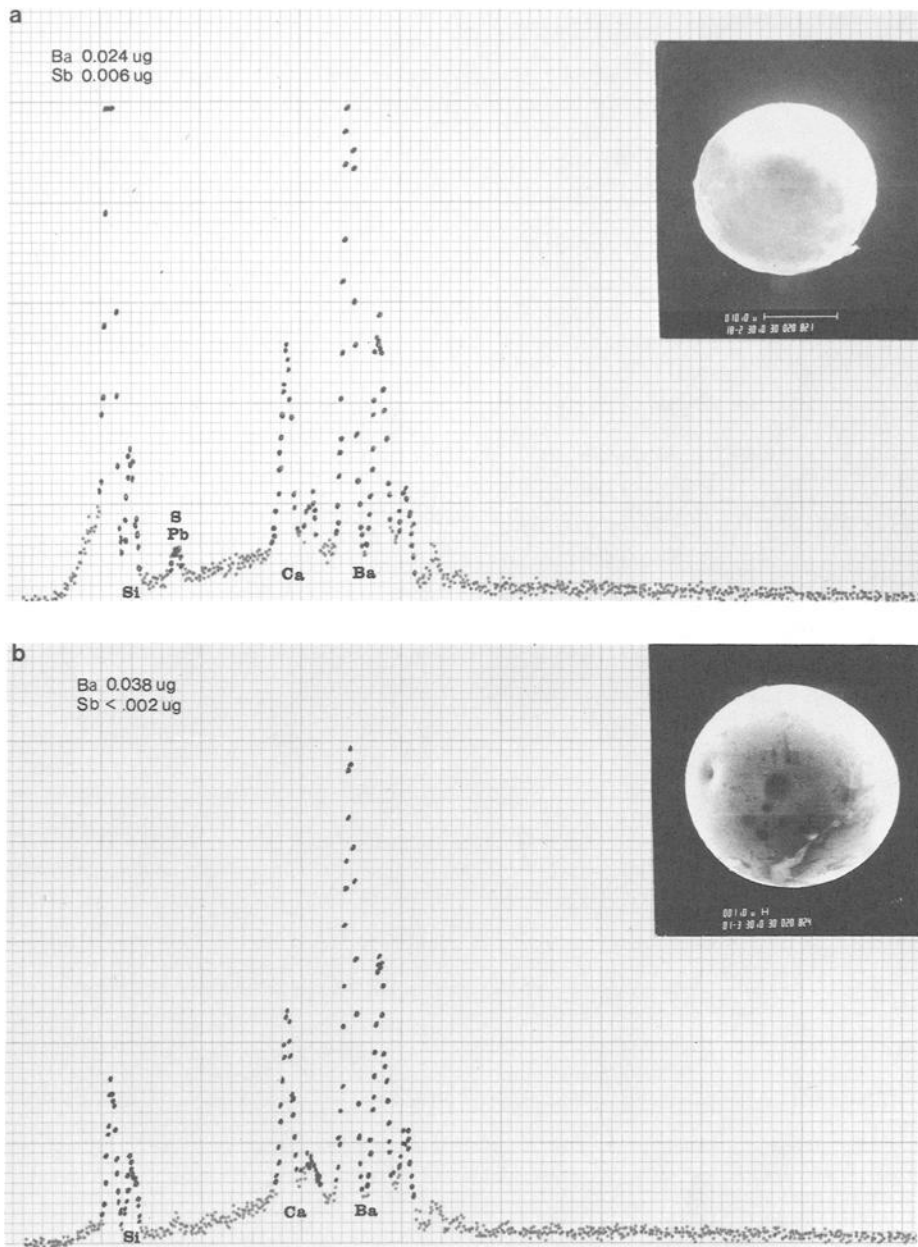


FIG. 5—Micrographs, EDX spectra, and NAA results from single particles of GSR.

GSR particles exist (for example, as a uniform contaminant layer on the hand), it is a minor factor.

The lack of reproducibility of particle counts from shot to shot (all else being held constant) was, at first, a surprise. But after the NAA data from swabs obtained from subsequent firings of the same weapon were examined it was found that the deposition may vary by as much as ten times [10], whereas in similar data collected in our laboratory [7]

the factor was roughly five. Hence the amount of deposition from a single firing of a gun has little meaning unless a variation of more than a factor of ten can be tolerated. Only averages of five to ten firings can produce any statistically meaningful results.

In the beginning of this work one of the items considered was the cleanliness of the gun prior to firing. However, the variation between single firings of a clean weapon versus the same weapon when dirty was smaller than the random variation from firing to firing. Thus no conclusion could be drawn without averaging. However, when examining the composition of GSR, one must bear in mind that the residue can be representative not only of the ammunition fired but also of previous ammunition that was fired since the gun was last cleaned.

To determine something about the homogeneity within the particles as well as their composition, particles were obtained from the primer cup of a spent Remington .45-caliber cartridge. One was fractured with a scalpel, and Fig. 6 is a composite micrograph showing

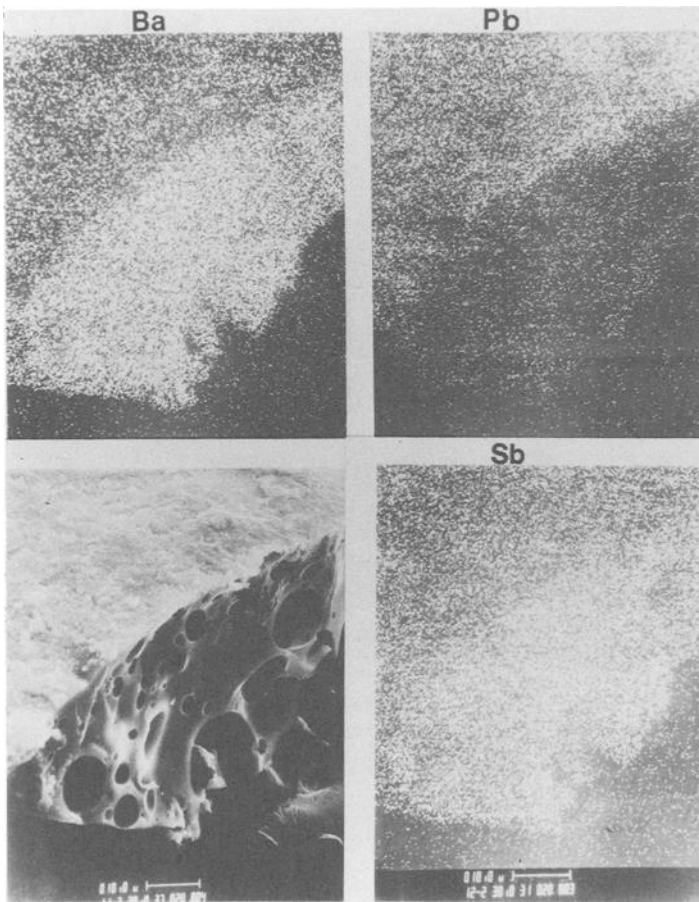


FIG. 6—Composite micrograph and elemental distribution from a GSR particle that has been fractured. The fractured surface is one having large perforations. The bar represents 10  $\mu\text{m}$ .

the particle (with its fractured surface) located in the lower left quadrant. The distribution of lead is seen to be mainly on the external surface of the particle while barium and calcium/antimony are distributed in the bulk (on the fractured surface). It should be emphasized, however, that this particle is much larger than those normally found on the

hands of a shooter. Another of the larger particles was not brittle (did not fracture). The cross section in this case was made up of lead globules. Thus the larger GSR particles seem to be varied agglomerates.

It was noted that some of the larger particles examined have surfaces covered with nodules. Figure 7 shows such a particle with the corresponding X-ray spectra from the

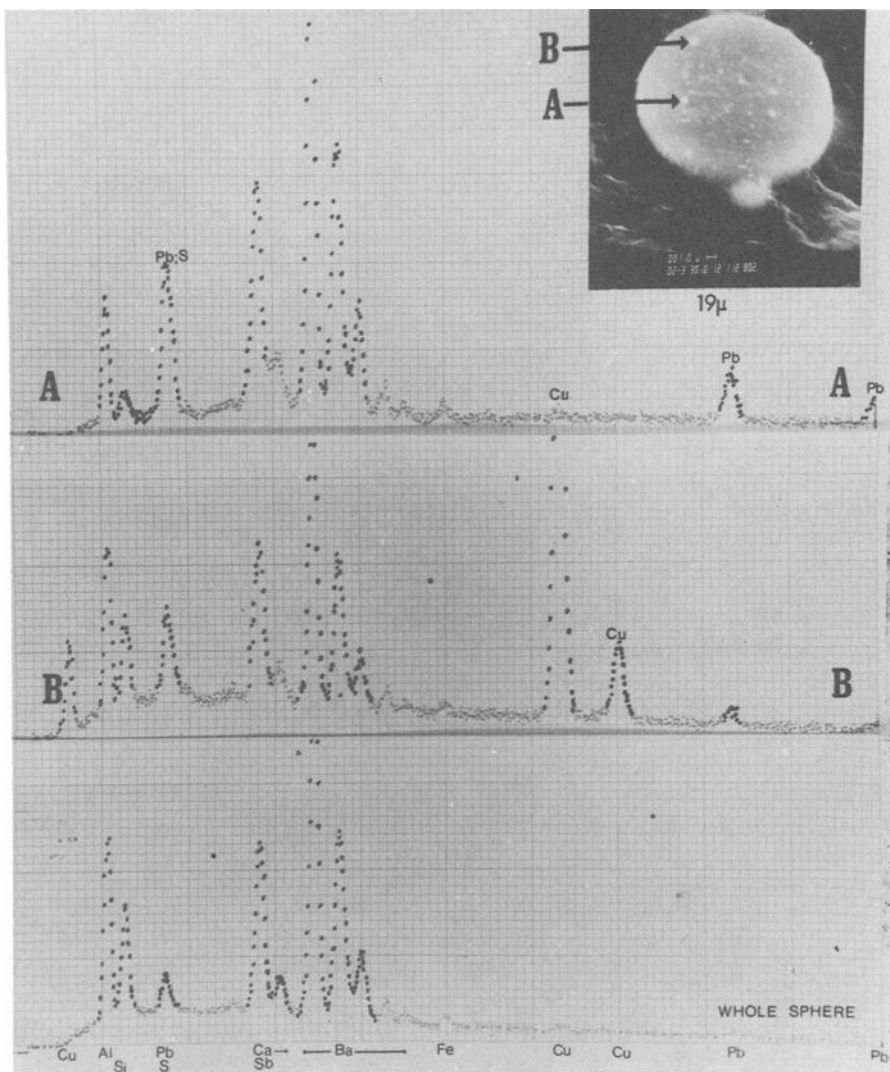


FIG. 7—Micrograph of GSR sphere having a bumpy surface and EDX spectra from individual nodules on the surface (A and B) and from the whole sphere.

whole sphere. However, when the electron beam is focused on Nodule A the lead peaks become very pronounced, indicating a high concentration of lead there. Most of the nodules showed a similar spectrum. The composition of Nodule B, however, seems to be higher in copper. Generally, lead appears to be associated with nodules, or rough-textured surfaces, in the larger spheroids.

Another experiment was performed to determine what particles, if any, were left suspended in the air after a shooting. One minute after the firing of the .38 revolver in a clean room, a piece of tape was allowed to collect particles for 1 min (the tape was moved around the room). The tape was found to contain 15 spherical lead particles per square millimetre with an average diameter of  $0.42 \mu\text{m}$ . Renfro and Jester [11] also detected suspended GSR in the air after 72 h. If a small number of submicrometre particles of lead are found on the hand of a suspect the examiner must consider the possibility that these could have been acquired by walking into a room after the shooting occurred. Of course they could also be the adherent remains of a more representative sampling of GSR that had been deposited on the hand at the time of the shooting.

## Discussion

The technique of identifying particulate GSR with the SEM and associated X-ray analyzer appears very promising. The efficacy of the technique in routine casework will depend on subsequent research and field trials.

The pros center on the possibility of collecting a few particles from the hands of a suspect that can be identified as originating from the discharge of a firearm. Thorough handblank studies have yet to be performed to determine the criteria required before an examiner can state unequivocally that the particles found are GSR. Once these background values are obtained and criteria for the presence of GSR particles are stated, an examiner can confirm the presence of GSR. After bulk analysis for antimony, barium, or lead by NAA or atomic absorption an examiner does not now have the ability of making as strong a statement because the possibility exists that the higher than usual amounts of antimony, barium, and lead could be from material other than GSR. This problem is especially serious when both hands have high concentrations of the elements of interest as compared to the mean handblank concentration.

The transfer of residue from the firing to the nonfiring hand would also cease to be a critical consideration if particulate analysis is performed because the conclusion is based on the presence of recognizable particles and not on the quantitative difference of barium, antimony, and lead between the two hands. The persistence studies that were performed by NAA [7] show that 6% of the barium deposited remaining on the hand after 5 h of normal activity. If the original amount deposited is about  $1 \mu\text{g}$  of barium the number of particles containing barium would be roughly 300 (not counting particles composed of lead only), assuming an average value of 3.3 ng of barium per particle. After the 5 h of delay, 18 barium-containing particles would be expected to be picked up by the tape, a number which lends itself to a search within a reasonable amount of time (1 h).

The cons associated with this technique are the method of collection and the time required for the search.

The method of collection with the sticky adhesive provides an unstable base for these particles. When the electron beam is focused on a particle, the volatile components of the tape are driven off by local heating, causing the surface to pucker or crack, or both, thereby displacing the particle. In addition, multiple dabbings of the hand may fully imbed into the tape adhesive some of the particles that were the first to be collected, thereby removing them from the counting process. This was tentatively concluded when some of the specimens placed in 5*N* hydrochloric acid for 5 min did not fully dissolve all the barium and antimony from the tape. An optimized method of collection and mounting should be developed to provide a stable substrate and a collection efficiency at least matching that of the adhesive layer.

The search for and identification of GSR particles from one case is a time-consuming process that now requires the full attention of an experienced operator over a time span ranging from 10 min to 8 h. This requirement is the technique's major shortcoming, not

only because of the man-hours involved but also because a distracted operator can produce false negatives. In our opinion, automation of the search process is one of the essential developments needed before this technique can be routinely used. We are now in the process of obtaining funds for implementing an automatic search for these particles that would remove most human subjective judgment.

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Elemental Analysis Unit  
 FBI Laboratory  
 Washington, D.C. 20535