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EQUIPMENT SYSTEMS IMPROVEMENT PROGRAM

FINAL REPORT ON PARTICLE ANALYSIS FOR GUNSHOT RESIDUE DETECTION

Law Enforcement Development Group

September 1977





Prepared for

National Institute of Law Enforcement and Criminal Justice LAW ENFORCEMENT ASSISTANCE ADMINISTRATION U.S. DEPARTMENT OF JUSTICE

The Aerospace Corporation (

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Prepared by

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Prepared for

National Institute of Law Enforcement and Criminal Justice LAW ENFORCEMENT ASSISTANCE ADMINISTRATION U.S. DEPARTMENT OF JUSTICE

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FINAL REPORT ON PARTICLE ANALYSIS FOR GUNSHOT RESIDUE DETECTION

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ABSTRACT

Since the possibility of using particle analysis for the detection of gunshot residue was first reported, ⁹ the background and operational characteristics of this new method have been firmly established and tested extensively in actual cases.

Particle analysis employs a scanning electron microscope, equipped with an x-ray analysis capability, to visually examine at high magnification gunshot residue removed from the hand by a simple adhesive lift technique. When viewed in this way, gunshot residue consists of discrete, micrometer-sized particles, predominantly spheroidal, and often of characteristic appearance. The x-ray analyzer will identify all chemical elements heavier than sodium that are contained in each individual particle. The only elements possible for gunshot residue are those that can be derived from the constitution of the bullet, a plating or jacket over the bullet, and ingredients of the primer. The morphology of the particles allows them to be readily found among the general debris (skin salts, minerals, and adhesive) lifted from the hand, and the chemical composition identifies them. Some particles are uniquely identifiable as gunshot residue by virtue of only their compositions. Other compositions are less unique but, when combined with morphological information, they are still typical of gunshot residue. Least characteristic are irregularshaped particles composed of lead only, but even these can usually be distinguished from particles from the exhaust of an automobile, which is the most common source of environmental lead contamination.

The ability to identify gunshot residue particles uniquely and to distinguish them from environmental sources of lead, barium, and antimony eliminates the threshold problem inherent in bulk elemental analysis. Although the amount of gunshot residue on the hand of a live subject declines rapidly with time, this independence from a quantitative restriction makes detection possible for up to 12 hours after a firing and accounts for the superior success rate of particle analysis when compared with previous methods.

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The collection of particles that constitute gunshot residue is further identifiable by a characteristic distribution of sizes, shapes, and compositions. These distributions have been determined completely for 24 cartridges, and partially for some additional representative cartridges. The resulting descriptions are found to differ for different families of cartridges, and to differ within a family with the muzzle velocity of the bullet. They differ very significantly between jacketed and non-jacketed bullets.

Farticles which individually resemble gunshot residue are produced by activities such as the firing of explosive rivets, the smelting of lead, and the manufacture of plates for lead-acid batteries. However, when taken as a whole, the residues from these activities contain many particles that are not consistent with gunshot residue in either composition or distribution. On this basis, the analysts who examined these occupational residues in blind tests never called them gunshot residue.

New experiments were performed to test the persistence of the particles on the hands of live subjects. Various calibers were used, and particle counts were compared with bulk elemental analyses. While the activity of the person can lead to considerable variation, the results confirm earlier experiments by others that, on the average, the loss during the first hour after the shooting is about one order of magnitude (a factor of ten). For a delay of ten hours, the loss averages two orders of magnitude (a factor of 100). Limited tests of the persistence on smooth fabric indicate similar behavior.

Samples for gunshot residue determinations from actual cases were solicited from law enforcement agencies. Eighty-six of the 100 cases analyzed that had been completed when this report was written were of a nature to lend themselves to statistical evaluations. In cases in which a handgun was used, gunshot residue was found in 90 percent of the cases. The delay between firing and sample collection for the positive results ranged from 1 to 13 hours, averaging 3 1/4 hours. Long guns were used in relatively few (15) cases, and residue was found in only 50 percent, with rifles accounting for a majority of the negative results and shotguns a majority of the positive results. In

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suicides, gunshot residue was detected as much as five days after the firing when the body was undisturbed. (There were no cases involving a longer delay.) However, handling and transportation of the body can lead to loss of residue, the degree of loss depending on the circumstances.

Detailed specifications have been established which will lead to the selection of a scanning electron microscope that is highly efficient for gunshot residue detection.

The hand-sampling procedure consists of pressing an adhesive-coated disk repeatedly against the areas of interest. The choice of the adhesive is the result of a number of compromises and provides a 70 to 75 percent collection efficiency.

The work accomplished in this project can be summed up by stating that sufficient background has been developed and enough practical experience with actual cases has been gained to consider the method to be ready for general use. The effectiveness of the method is extremely high for residue from handguns, and even for long guns it is higher than that of previous methods.



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PREFACE (For Criminalists and SEM Analysts)

This document is more than a report on work accomplished. It is conceived by its authors to be also a textbook and a manual for the particle analysis method of gunshot residue detection by criminalists and scanning electron microscope (SEM) operators. It is planned to provide all the information that is presently available to perform the analysis successfully, interpret the results, and explain and defend them in court.

This report does not contain instructions in the basic technique of operating the SEM, which is not very complicated. When the SEM is purchased, manufacturer's representatives train the operator well enough to enable him to operate the instrument. However, there are books, workshops, and university courses available to permit an operator to acquire the advanced techniques needed to get the most out of the instrument and out of diverse types of specimens.

This report cannot impart experience to criminalists and SEM operators. Knowing the basics of electron microscopy, and having studied this document, gunshot residue determinations can be made. However, initially they will be accomplished slowly. Several weeks of full-time experience working with the SEM are required to gain the proficiency needed to quickly discriminate between possible gunshot residue particles and particles of other origins, and to quickly recognize x-ray peaks without referring to tables. Particle analysis is labor-intensive and the skills of criminalists and SEM operators can be brought to a peak by constant practice with this instrument. Criminalists are highly versatile individuals who normally do many different things during a working day. Many criminalists may therefore be unaccustomed to this mode of operation. However, a laboratory that is large enough to afford a SEM is also likely to have a workload that will keep it busy throughout the working day. A permanently assigned skilled operator is required to get the most out of the SEM and to keep it in good working order.

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Special thanks are due to J. F. Anderson, Director of the Eastern Washington State Crime Laboratory, for his efforts in obtaining occupational handsamples from a lead smelting plant in Idaho, to G. Gonzales of the Orange County, California, Sheriff's Crime Laboratory for providing samples from battery plate assemblers, and to M. J. Camp of Northeastern University and R. Saferstein of the New Jersey State Police Forensic Science Bureau for miscellaneous additional samples.

The results reported in this document are built on a foundation comprised of earlier work by some of the authors of this report as well as by other members of the staff, especially J. E. Wessel.

SUMMARY

This section is an "Executive Summary" which is intended to be read by executives who need to know the concepts developed and major conclusions reached, but who do not require the extensive technical and procedural detail needed by those readers who may practice the methods described or interpret them in court. Other readers may proceed directly to Chapter I.

The particle analysis method of gunshot residue detection was developed by The Aerospace Corporation under a contract with the National Institute of Law Enforcement and Criminal Justice, the research branch of the Law Enforcement Assistance Administration. The contract began in January 1974 and ended in September 1977. The first tasks completed under this contract were a comprehensive survey of crime laboratory practice and a thorough assessment of the then existing state of the art and any technology relevant to the forensic detection of gunshot residue on the hands of individuals. The eventual outcome of the technology assessment was a three-pronged experimental program toward the development of more effective detection methods. Two of these, a search for organic residues of smokeless powder and a photoluminescence method for the bulk analysis of lead and antimony, are described in a number of separate reports, as is the original survey and assessment. This document is the final report on the third approach, particle analysis.

Particle analysis employs a scanning electron microscope, equipped with an x-ray analysis capability, to visually examine at high magnification gunshot residue removed from the hand by a simple adhesive lift technique. When viewed in this way, gunshot residue consists of discrete, micrometer -sized particles, predominantly spheroidal, and often of characteristic appearance. The x-ray analyzer will identify all chemical elements heavier than sodium contained in each individual particle, and the only elements possible for gunshot residue are those that can be derived from the constitution of the bullet, a plating or jacket over the bullet, and ingredients of the primer. The morphology of the particles allows them to be readily found among the general debris (skin, salts, minerals, and adhesive) lifted from the hand, and the chemical composition identifies them. Some particles are uniquely identifiable as gunshot residue by virtue of their compositions alone. Other compositions are less unique but, when combined with morphological information, are still typical of gunshot residue. Least characteristic are irregular-shaped particles composed of lead only, but even these can usually be distinguished from particles from automobile exhaust which is the most common source of environmental lead contamination. Most automobile exhaust particles contain bromine, and very few are spheroidal. The scanning electron microscope x-ray analyzer routinely identifies all elements present including bromine. Neutron activation or atomic absorption analyses identify only the elements specifically looked for, and they cannot analyze for bromine without extensive chemical processing of the sample.

The ability to identify gunshot residue particles uniquely and to distinguish them from environmental sources of lead, barium, and antimony eliminates the threshold problem inherent in bulk elemental analysis. Although the amount of gunshot residue on the hand of a live subject declines rapidly with time, this independence from a quantitative restriction makes detection possible for up to 12 hours after a firing and accounts for the superior success rate of particle analysis when compared with previous methods.

Some particles have been termed typical but not unique. There is a fairly limited number of activities, principally the firing of explosive rivets, the smelting of lead, and the manufacture of plates for lead - acid batteries, that produce particles which individually resemble gunshot residue. When taken as a whole, however, the residues from these activities contain many particles that are not consistent with gunshot residue in either composition or distribution. On this basis, the analysts who examined these occupational residues in blind tests, never called them gunshot residue.

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The collection of particles that constitute gunshot residue is further identifiable by a characteristic distribution of sizes, shapes and compositions. These distributions have been determined completely for 24 and partially for some additional representative cartridges. The resulting descriptions are found to differ for different families of cartridges, and within a family with the muzzle velocity of the bullet. They differ very significantly between jacketed and non-jacketed bullets. On the other hand, the presence of only a plating on the bullet introduces copper and sometimes zinc into the residue but does not change the distributions found for similar cartridges with bare lead bullets.

For each broad class of cartridges, for example, all . 22s as one class, all .38 specials with lead or plated bullets as another, and all jacketed . 38 specials as a third, there is a straight-line relationship between the logarithm of the number of particles and the muzzle velocity of the bullet. The higher the velocity, the lower the particle count. Cartridges giving the largest numbers of particles, and therefore giving the residue that is easiest to find or can be found for the longest time after firing, are the common 40 grain .22 caliber bullets and the plain lead round-nose or wadcutter bullets of , 38 special caliber, shot from shortbarrelled guns. Much lower amounts of residue result from fast, jacketed bullets, such as 9mm caliber, or the lightweight .38 special hollow-points. For the .22 rimfire cartridges, there is a modest but regular increase in the proportion of primer to bullet particles and of irregular to spheroidal particles with increasing velocity. For the .38 and similar caliber ammunitions, these trends are less obvious, but there is a major difference in the proportions when jacketed and non-jacketed bullets are compared. In general, high-velocity cartridges produce larger particles and a greater proportion of irregular particles than cartridges with low bullet velocities.

New experiments were performed to test the persistence of the particles on the hands of live subjects. Various calibers were used, and particle counts were compared with bulk elemental analyses. While the activity of the person can lead to considerable variation, the results confirm earlier experiments by others that, on the average, the loss during the first hour after the shooting is about one order of magnitude (a factor of ten). For a delay of ten hours, the loss averages two orders of magnitude (a factor of 100). Limited tests of the persistence on smooth fabric indicate similar behavior. Fluffy or deep-pile fabrics were not tested because of excessive fiber pick-up in the adhesive collection method. Bystanders, standing three or ten feet (about one or three meters) away and abreast of the shooter, had gunshot residue on their hands at the 3-foot distance, but not at the 10-foot distance.

During the final year of the contract, samples for gunshot residue determinations from actual cases were solicited from law enforcement agencies. Eighty six of the 100 case analyses that had been completed when this report was written were of a nature to lend themselves to statistical evaluations. In cases in which a handgun was used, gunshot residue was found in 90 percent of the cases. The delay between firing and sample collection for the positive results ranged from 1 to 13 hours, averaging 3-1/4 hours. Long guns were used in relatively few (15) cases, and residue was found in only 50 percent of the cases, with rifles accounting for a majority of the negative results and shotguns for a majority of the positive results. In suicides, gunshot residue was detected as much as five days after the firing when the body was undisturbed (there were no cases involving a longer delay). However, handling and transportation of the body can lead to loss of residue, the degree of loss depending on the circumstances.

In order to perform particle analysis for gunshot residue detection efficiently, the scanning electron microscope must meet certain detailed specifications and must have some specific features. The availability of these features is not, however, a function of price; some relatively low-cost instruments are suitable. The instrument specifications depend mainly on the manufacturers' concepts of their principal markets. Therefore, a thorough study should be made before purchasing an instrument. The selection of the x-ray system allows a greater degree of latitude.

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The hand-sampling procedure consists of pressing an adhesive-coated disk repeatedly against the areas of interest, going from point to point in a regular pattern. The choice of the adhesive is the result of a number of compromises and provides from 70 to 75 percent collection efficiency at present. The disk is designed for direct insertion into the scanning electron microscope. The collected particles were found to be distributed randomly over the sample surface, with a fair degree of uniformity. Statistical calculations based on this distribution pattern and the geometry of the search pattern employed provide the result that examination of 15 to 20 percent of the sample area reduces the probability of not finding at least a few particles to a low value if residue is present.

Similar calculations for more ample residues also show that examination of 10 to 15 percent of the sample area is sufficient to allow valid extrapolations to the number of particles and their size and compositional distributions in the entire sample.

The work can be summed up by stating that sufficient background has been developed and enough practical experience with actual cases has been gained to consider the method to be field-ready. The effectiveness of the method is extremely high for residue from handguns, and even for long guns it is higher than previous methods.

An experienced operator can complete examinations for two average cases in one eight-hour day. For large service laboratories with a heavy workload, it may be desirable to automate the procedures. This would require a considerable engineering effort, but there seem to be no major technical obstacles that would prevent automation from being accomplished.

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CHAPTER I. BACKGROUND AND SUMMARY OF PROJECT HISTORY

A. General.

The Aerospace Corporation has been under contract to the National Institute of Law Enforcement and Criminal Justice (NILECJ), the research branch of the Law Enforcement Assistance Administration (LEAA) since January 1974, in a program to develop improved methods for the detection of gunshot residue on a person's hands. The ultimate objective of the program was to develop a method or methods that would provide positive identification of gunshot residue which would be effective for several hours after a shooting, as well as being reasonably fast and inexpensive.

The initial tasks completed under this program were (1) a detailed survey of the literature bearing on the problem and of the views, requirements, case loads, and practices of crime laboratories, (2) an assessment of the then prevailing state of the art, and (3) an extensive examination of analytical technology for the identification of concepts that may lead to improved detection methods for gunshot residue, and the selection of some of them for further study. A comprehensive report was issued, describing this work.¹ This was followed by three separate development programs in 1) bulk elemental analysis for lead and antimony by photoluminescence, 2) detection of organic components of smokeless powder, and 3) particle analysis by scanning electron microscopy.

This report is devoted to particle analysis. However, the other two approaches are also summarized in this chapter in order to put particle analysis in the proper perspective and to provide an overview of the total program for the reader.

At the time the survey was conducted, the only method that had a measure of acceptance was neutron activation analysis for barium and antimony, which however was in the process of being rivaled and partly supplanted by atomic absorption spectroscopy for lead, barium, and antimony. Both methods belong to the category of bulk elemental analysis schemes, in which the total quantities of the elements named that were removed from someone's hand by various collection methods are measured. This type of

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analysis cannot distinguish between different sources of the elements, whether they form a continuous film or discrete particles, and how the elements are distributed in either of the foregoing.

Lead and barium are common environmental contaminants. While antimony is less widely distributed, certain occupations give rise to high levels of this element (electronic assemblers, auto mechanics, painters, and machinists)². In order to allow for this environmental background of these elements, thresholds were suggested which had to be exceeded in order to indicate that the person examined may have fired a gun. For example, the Treasury's Bureau of Alcohol, Tobacco, and Firearms suggested thresholds of 0.3 μ g of barium (Ba) and 0.2 μ g of antimony (Sb). Lead (Pb) is not determined in neutron activation analysis because of low sensitivity, but can be determined by atomic absorption spectroscopy. However, additional equipment and procedures are required to do this, since each element is determined separately. The neutron activation procedure suffered from excessive turn-around time, because samples had to be sent to a nuclear reactor facility for analysis. Atomic absorption provided results much more quickly because it could be carried out locally by those larger laboratories that were able to acquire the necessary equipment.

No matter how it was done, bulk elemental analysis suffered from two deficiencies. In experiments in which residue was collected promptly after firing a gun, just slightly over one-half the .38 caliber firings and only about one-fifth the .22 caliber firings give residues that equalled or exceeded the thresholds. Figure 1 shows the range of antimony levels for many such firings as determined at the Gulf General Atomic Corporation.² The second deficiency is the rapid decline of these levels with the passage of time, as shown in Figure 1, using data determined by Kilty for a .45 caliber³ and by Aerospace for a .32 caliber handgun.⁴ Since in actual cases some time always passes between the shooting and the sampling of a suspect, it is readily understandable that the success rate of elemental analysis was quite unsatisfactory.

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Figure 1. Antimony Levels on the Firing Hand

The detection of much higher amounts of the critical elements has been reported, 7 apparently as the result of changes in the collection and sample work-up procedures.

The rapid loss of residue from the hand may not be experienced on immobile subjects such as suicide victims, at least not if the victim remains undisturbed. The success rate will then depend more on whether the ammunition used deposited larger or smaller amounts of residue. The literature^{2, 4, 5} shows a wide range for the amounts of Pb, Ba, and Sb found in prompt collections and determined by bulk elemental analysis. Recently, five medical examiners' laboratories have cooperated with The Aerospace Corporation in a new field test of elemental analysis for suicide investigations. The method used in this field test is an Aerospace-developed photoluminescence procedure for the essentially simultaneous determination of lead and antimony^{4, 5} which involves substantially lower equipment costs and is fast and relatively simple. The objectives of this study were: (a) to obtain more statistics for the evaluation of bulk elemental analysis in general and photoluminescence in particular as a tool for investigating suicides, and (b) to obtain new background data for possible updating of threshold recommendations.

The study of analysis by photoluminescence was initiated by The Aerospace Corporation in 1973 under a company-financed research program in forensic science and was subsequently incorporated into the work performed for the LEAA. It was brought to completion with the field test in which levels of lead and antimony were determined on the hands of many suicide victims and compared with the levels of these elements found on the hands of persons who died of other causes. The field test and the results obtained in the field test are the subject of a separate report.⁶

The Aerospace Corporation has pursued two additional approach... to improved detection methods for gunshot residue. One of these was based on the concept that organic compounds characteristic of smokeless powder may be detectable on the hand that fired a gun. The work that evolved out of this concept is described in a companion report.⁸ The first part of that

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report describes the analysis of 32 samples of smokeless powder which were removed from commercial cartridges, and of one reloading powder. These were analyzed for their volatile organic components by computerized gas chromatography-mass spectrometry. The second part of the report describes the analysis of actual gunshot residue samples to see whether any of the previously identified compounds or any derivatives formed as a result of the firing can be detected. The efficiency of the solvent-swab technique that was used was also investigated. It was found that the volatile organic compounds that were present in the original powder could be detected solely as constituents of powder flakes recovered from the hand or from the muzzle blast.

Flakes of smokeless powder, ranging in condition from slightly to severely burned, are often seen in promptly collected residue but are very scarce when collection is delayed. Based on these observations, the collection of statistics on the occurrence of such flakes on the hands of suicide victims was made part of the luminescence field test. A very inexpensive procedure has been developed whereby characteristic organic compounds in powder flakes can be identified by thin layer chromatography to positively confirm the nature of the flakes. Discrimination between paint and explosive grades of nitrocellulose is part of this test, so that it can be used even for single-base powders.

The final approach to an improved detection method led to the development of particle analysis, which gives the most positive identification and has had the highest success rate in actual cases. It is the main topic of this report. Through the use of a scanning electron microscope (SEM) equipped with an x-ray analyzer, it was discovered that the characteristic elements, lead, barium, and antimony, as well as other bullet and cartridge components, are mostly (if not entirely) contained in discrete particles, the majority of which have characteristic spheroidal shapes. This morphology and the relative brightness of the particles make it possible to find them among the background material removed from the hand by a simple adhesive lift technique developed for this purpose. This collection

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technique was also found applicable to the foregoing luminescence analysis. The crucial difference between particle analysis and bulk elemental analysis is that the former provides two additional items of information not obtainable with the latter. One is the morphology of the particles, and the second is a complete and simultaneous identification of all the elements present in a particular particle instead of the quantity of a few selected elements in the entire sample. The sum of this information leads to a positive identification of gunshot residue, which is independent of the quantity present, thus circumventing the threshold problem. Since gunshot residue can be distinguished from most other sources of the same elements (as an example, from automobile exhaust), it is not necessary that a certain quantity be exceeded. Thus, gunshot residue can be found and identified for several hours after a firing despite the rapid decline of the quantity with time.

The ability to recognize gunshot residue in all samples collected promptly and in many collected some hours later has been confirmed in several hundred test firings, while at the same time control samples taken from the hands of persons who had not fired a gun, never resulted in a false positive indication. These results were first reported in December 1974.⁹

In October of 1975, a demonstration-familiarization seminar on gunshot residue detection was held at The Aerospace Corporation, which was attended by about twenty criminalists from the United States and Canada. All aspects of the problem were explored, and the particle analysis method was demonstrated and tested in both known and blind experiments. The minutes of this seminar were published in a report¹⁰ which has received only limited distribution because it was primarily intended to provide the participants with a record of the proceedings.

One of the major conclusions reached by the participants and concurred in by the Aerospace personnel involved, was that Aerospace was

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successful in identifying gunshot residue but could not adequately describe the criteria by which particles were judged to be gunshot residue: The operators were experienced and successful, but they practiced an art that had not yet become a science. What was lacking was a clear definition, usable by others, of what constituted gunshot residue, and a major effort toward the objective characterization of gunshot residue was recommended. An intensified exploration of environmental and occupational particles that might resemble gunshot residue sufficiently to require criteria for differentiation was also recommended. These two tasks were initiated promptly and recently completed.

These characterization studies of gunshot residue and of occupational residues are described fully in the current report with respect to both methodology and results. Appendix D of this report comprises a collection of photographs of particles of interest.

In January of 1977, The Aerospace Corporation initiated the final phase in the development of particle analysis to ensure its field readiness. This task was carried out concurrent with the continuing characterization studies and consisted of case assistance to law enforcement agencies free of charge. The objectives of the case work were:

- To detect and solve any remaining problems that may be encountered in actual cases,
- To speed the transfer of the method from the laboratory to field use, and
- To acquaint more criminalists with the details of the method well enough to start them practicing it.

Slightly over 100 gunshot residue determinations have been performed under the case assistance program. Positive results were obtained in 90% of the cases that involved the use of a handgun and in 50% of the small number of cases that involved long guns.

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Although the work reported here is presently the most comprehensive study of the application of scanning electron microscopy to gunshot residue determination, there have been others. Boehm¹¹ and Diederichs et al.¹² have independently published brief accounts, and so has Keeley.¹³ Andrasko and Maehly¹⁴ have reported some findings which are in general agreement with those reported in this report. Matricardi and Kilty¹⁵ of the FBI laboratory have recently published a comprehensive and interesting independent investigation of particle analysis for gunshot residue determination.

B. Scanning Electron Microscope (SEM) Operation.

The principle of the SEM is illustrated in Figure 2. A beam of electrons is scanned over the surface of a specimen, causing secondary electrons to be emitted. The intensity of the secondary electron emission depends on the topography (hills & valleys) of the surface as well as its composition, and hence can be used to build up a point-by-point image of the surface, as the primary beam is scanned over the specimen. Because the primary electron beam can be very finely collimated, and because high-energy electrons have an equivalent wave-length much shorter than that of visible light, the electron microscope allows magnifications to be achieved that are much greater than that of the optical microscope. The usual working magnification of an optical microscope ranges up to 1000 times, with 2000 being an upper limit. Working magnifications in the scanning electron microscope are from ten to one hundred times greater, and special research instruments can do even better. Also, the SEM has an extraordinary depth of focus so that the tops of peaks and the bottoms of valleys in a surface can be seen in focus simultaneously, unlike the optical microscope which must be refocused for slight changes in elevation.

Another feature of the SEM, which has no analogy in the optical microscope, is that the primary electron beam causes x-rays to be

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Figure 2. Scanning Electron Microscope with X-ray Detection

emitted by the specimen. The energy of these x-rays is characteristic of the chemical elements that are present in the specimen. This makes it possible to obtain a chemical analysis of the area under observation.

Figure 3 illustrates how particles may be recognized. The distinctive morphology of the particle shown on top suggests that it may be gunshot residue. This is confirmed by the presence of all three of the elements, lead, barium, and antimony within this particle, as shown by the x-ray spectrum to the right of the picture. The lower picture is that of an automobile exhaust particle. It lacks the characteristic spheroidal shape of the other. Its origin is uniquely established by the simultaneous occurrence of lead and bromine within this particle.

PARTICLE ANALYSIS LEADS TO POSITIVE IDENTIFICATION BECAUSE IT DISTINGUISHES BETWEEN SOURCES BY MORPHOLOGY AS WELL AS COMPOSITION



GUNSHOT RESIDUE PARTICLE 3.5μm







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CHAPTER II. RESIDUE CHARACTERISTICS AND CLASSIFICATION OF PARTICLES

A. Composition of Gunshot Residue Particles.

1. <u>Elements present in cartridge components</u>. All of the elements detectable in gunshot residue, with the exception of skin salts and iron which can arise from wear of the gun, must originate in some component of the cartridge. Therefore, existing information on the composition of cartridges is reviewed in the following paragraphs.

Bullets are made of lead, usually with a small amount of antimony. This is discussed in III. B. 2 of this report. If present, a plating on the bullet may be nearly pure copper, Lubaloy, or, less commonly, yellow brass. Lubaloy is a trade name designating a plating alloy of 90 percent copper, 8 percent zinc, and 2 percent tin. * It is essentially a member of the red brass family, which runs from 90 to 95 percent copper and from 5 to 10 percent zinc. Yellow brasses have a 30 to 35 percent zinc content and may contain traces of lead and iron.

Jackets are also made of copper, red brass, or yellow brass. The addition of a few percent of tin to red brass for bullet jackets is obsolete, as is the use of cupro-nickel, which is a copper alloy that contains from 10 to 30 percent nickel and about 1 percent iron.

Armor-piercing ammunition has both a steel core within the lead bullet and a steel jacket. For handgun ammunition, brass-plated jackets of mild steel are obsolete, but may still be encountered occasionally.

Black powder contains potassium nitrate, sulfur, and charcoal. Potassium and sulfur, but not carbon and nitrogen, are detectable by SEM/ x-ray analysis, but are not characteristic enough to be useful. Except for

*Tin is not usually detectable at this concentration.

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some calcium, modern smokeless powder contains only light elements which are not detectable by the type of x-ray analysis used in this work.

The shell or cartridge case is yellow brass, often nickel-plated. The last and most complex component of the cartridge is the primer mixture. It may contain some purely organic compounds which do not contain x-ray detectable elements, but it nearly always contains substantial amounts of lead styphnate and sometimes other lead compounds. Except for .22 caliber cartridges, it seems that all U.S. and Western European primers less than 15 years old contain both barium and antimony. Eastern European amunition exported to the Middle East is said¹⁶ to lack barium. However, the residues from test firing a .22 LR Valor brand cartridge, imported from Yugoslavia and submitted in a case, were rich in both barium and antimony.

The rimfire primers for .22 caliber ammunition also contain barium, except for Remington and Remington-Peters brands, but only Federal and some German and Austrian makes contain antimony.¹⁴ Silicon is present in most primers for all calibers. G. R. Warren^a has written that some primer mixtures are said to contain Prussian Blue, an iron-containing pigment, and that another mixture is supposed to contain copper thiocyanate. The antimony is always added as antimony sulfide, and other sulfides may be used as well. The use of powdared metals, such as magnesium, titanium, zirconium, and aluminum has been mentioned, but only aluminum appears to be common. The latter has been detected repeatedly in residue particles and in some unfired primers.

Many propile in the field believe that manufacturers do considerable experimentation with primer compositions, so that any specific information about these compositions must be considered subject to possible change at any time.

^aG. R. Warren, Eastern Washington State Crime Lab., Private Communication.

Potassium, chlorine, and occasionally traces of phosphorus are found in residue particles, but also appear in particles of non-residue origin. They are not present in primer mixtures, but are probably absorbed onto the particles from various salts on the skin. The old corrosive primers, which were phased out in the Western World more than 15 years ago, contained potassium chlorate, and mercury and tin compounds.

2. <u>Compositional criteria for identification</u>. The identification of a particle as either gunshot residue or as "consistent with gunshot residue" rests on a combination of morphological and compositional criteria, and also on the association of the particle with other particles, judged in the light of the distributions of sizes, shapes, and compositions recorded in subsequent sections of this chapter and Chapter V.

Compositional criteria are described in this section. The following three compositions have thus far been observed only in gunshot residue and are therefore considered characteristic.

- Pb, Sb, Ba.
- Ba, Ca, Si, with a trace of S.
- Sb, Ba.

Any particle having one of these compositions may also contain one or several of the following and only the following elements: Si, Ca, Al, Cu, Fe, S, P(rare), Zn(only if Cu > Zn), Ni(rare, only with Cu, Zn), K, Cl. The occurrence of some Sn is a possibility in obsolete ammunition.

The following compositions are consistent with gunshot residue, but are not unique to it:

- Pb, Sb.
- Pb, Ba.
- Pb.
- Ba if S is absent or only a trace.
- Sb(rare).

Any of the additional elements listed above, and only those, may again be present. The first two compositions, although not unique, have been found in few occupational residues (Chapter V). They are thus fairly characteristic although not conclusive.

The compositions, shapes and sizes of a collection of particles found in a sample should fall into a pattern that is consistent with the distributions described in later sections of this chapter. Furthermore, particles that are consistent with gunshot residue individually should not be found in the company of otherwise similar particles that are inconsistent with gunshot residue (see Chapter V).

For example, an ample deposit of only the second category of particles, those that are consistent with but not unique to gunshot residue, would be considered to be gunshot residue if there are no particles in this deposit that are not consistent with gunshot residue or have a clearly identifiable separate origin. Particles with a clearly identifiable separate origin may include particles that can be positively identified as automobile exhaust or as lighter flint residue (Chapter V). In the presence of inconsistent particles that are not clearly identifiable in this manner, the entire sample is rejected as evidence for gunshot residue. If only so few consistent (and no other) particles are found that one cannot know whether or not they are typical of the presumably larger population originally present, then the only finding that can be issued is that some particles have been found that are consistent with but not unique to gunshot residue. Such a finding can still be of value in those cases where the only question to be answered is a choice between one suspect or another.

3. <u>Bullet and primer particles</u>. Cartridges with bare lead bullets give residues that contain a large excess of lead-only particles (or leadplus-copper particles if the bullet is plated). Jacketed bullets give substantially fewer lead-only or lead-plus-copper particles. For this reason, it is inferred that most of the lead particles are derived from the bullet and, in this report, are classified as bullet particles, provided that in addition to

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lead, they contain only elements that can come from a plating or jacket, and provided that they contain no more than a trace of antimony.^a

Particles which contain barium, antimony, or silicon among their major or minor constituents, as defined in Chapter IX, are classified as primer particles.

These definitions tend to somewhat underestimate the number of particles actually derived from the primer. Primers also contain lead, and since primer residue is very heterogeneous, a few pure lead particles could originate in the primer.

The simple division into bullet and primer particles is highly useful for descriptive purposes, but it should be understood to be arbitrary. Most of the spheroidal (see the following section) particles are thought to arise by condensation from the vapor state. If given sufficient time, all vapors will mix thoroughly. The heterogeneous nature of residue indicates that vaporization and condensation take place much too rapidly for complete mixing to occur. Furthermore, vaporization of primer material and bullet material probably does not occur at precisely the same time. Nevertheless, there is bound to be some mixing, and some particles will receive contributions from more than one source. That this is true is indicated by the fact that material from bullet platings and jackets can be found both in bullet and in primer particles.

B. Morphology and Size.

The four types of objects found in gunshot residue are described in the following paragraphs. In the majority of cases, 70 to 100 percent of the particles in a sample of gunshot residue are spheroidal. These may be perfect spheres, or they may be stretched, dented, or otherwise distorted, but "three-dimensional roundedness" is a characteristic of this classification.

^aTrace is defined in Chapter IX, and refers to an amount that is "just detectable".

The surfaces of the spheroids may be smooth or fuzzy, scaly, or covered with smaller spheres. Occasionally, they are capped, perforated, broken, or stemmed. A few examples are shown in Figure 4. A more complete picture atlas is contained in Appendix D of this report.

The vast majority of the spheroidal particles have diameters of less than 5 µm. For practical reasons, no attempt is made to detect and count particles less than one-half μm in diameter. When characterizing a particular residue, one counts the number of particles in each of several size ranges. Because the numbers decline drastically with increasing size, the larger size ranges are chosen to be wider through the use of a logarithmic scale with base 4 for the diameters. Each size range is four times as wide as the next smaller one, and its midpoint is also a diameter four times as large as that of the next smaller range. The resulting scale is as follows:

DESIGNATION	RAN (rE (µm)	MIDPOINT (µm)
Α	0.5 - 2.0	1.25
В	2.0 - 8.0	5.00
С	8.0 - 32.0	20.00
D	> 32	_

Most of the remaining gunshot residue particles (rarely more than 30%, depending on the ammunition) are irregular. These are particles that have the same compositions as the spheroidal particles, but do not share the spheroidal or globular shape. They are irregular fragments, not infrequently somewhat flattened and flaky looking. It is important to note that they do not exhibit any features suggestive of crystal fragments, there are no crystal faces or edges. Gunshot residue is not visibly crystalline, although diffraction methods (x-ray or electron diffraction) do give crystal patterns (see Appendix A). The sizes of the irregular particles vary over a wide range, from less than one to several hundred µm. Because quite often they are few in number, no statistics concerning their size distributions



1.2 µm, Pb .22 COLT, 6 in., LR FED POWERFLITE, 40 gr RNL

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20 µm, Pb, Sb, Ba, (S) .380 BROWNING STANDARD SUPERVEL, 88 gr JHP

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11.6 µm. Ba. Ca. Si, Pb .32 LLAMA BROWNING, 71 gr FMJ

29µm, Ba, Si, Ca, (Fe) .32 LLAMA BROWNING, 71 gr FMJ



14 µm, Ba, S, Sb, (Pb) .38 SMITH & WESSON, 2 in., SPECIAL W-W, 148 gr WC

Figure 4. Examples of Spheroidal Particles



53µm, Ba, Ca, Sb, Si, Pb, Cu .38 SMITH & WESSON, 4 in., SPECIAL REM, 125 gr SJHP

have been taken. Like the spheroidal particles, their compositions divide them into bullet and primer particles. While this information has been recorded, it is not quoted in this report. Sometimes the ratio of irregular bullet to primer particles is similar to that of the spheroidal particles, and sometimes it is not. However, because of the frequently small numbers involved, these ratios may not be meaningful.

Despite the non-descript (our original term for them) appearance of the irregular particles, experienced operators have learned to recognize them. Sometimes, they have distinguishing features that are obvious. For example, cracks running through the particles are rather characteristic. The larger of the irregular particles often have several of the small spheroidal particles attached to them, and this is highly characteristic. Figure 5 shows some irregular particles.

A third type of particle of very limited occurrence is the cluster. This is an object that consists of from five to several hundred spheroidal particles attached to one another, somewhat like a bunch of grapes. Sometimes, the cluster is further attached to an irregular particle as if on a tray, or an irregular particle runs through the center of the cluster. They have been observed with some frequency in residue from 9 mm cartridges, less often from .357 magnum, and quite infrequently from .38 special cartridges. Clusters appear to be primarily a product of high power or high velocity.

The final type of object that may occur in gunshot residue is a fragment of smokeless powder. It is always referred to as a flake in this report to set it apart from the particles, which are of an inorganic nature. The flakes range in size from micrometers to almost a millimeter. They are few in number and are mostly seen in residue collected immediately after firing. Quite often, a few spheroidal particles are embedded in the surface of a flake.



 $82 \ x \ 60 \ \mu\text{m}, Pb, Ba, Sb, Si$.22 COLT, 6 in. FED. POWERFLITE, 40 gr RNL



60 x 160 μ m, Ba, Pb, Si, Ca, Sb .32 LLAMA BROWNING, 71 gr FMJ



335 x 110 µm, Ba, Sb, S, Pb .380 BROWNING SUPERVEL, 88 gr. JHP



52 x 35 μm, Pb, Ba (Sí, Sb) .22 COLT, 6 in., LR FED. POWERFLITE, 40 gr RNL



100 x 50 (larger), Ba, Si, Ca, Pb 9mm BROWNING, HI-POWER FED. 123 gr FMJ



500 x 500 μm 9mm BROWNING, Hi-POWER SPEER, 100 gr JHP

Figure 5. Irregular Particles (first four), Cluster (bottom, middle), and a Flake (bottom, right)

The spheroidal particles are thought to result by rapid condensation from a vapor, whereas the irregular particles may be produced by the solidification of droplets of molten material that were flung against the inside surfaces of the gun.

CHAPTER III. CHARACTERIZATION STUDIES

A. Velocity Measurements.

Except where noted, measurements of muzzle velocities and residue characterizations were performed using cartridges from the same box of ammunition. A Chronotach instrument^a was used, with Polyscreens spaced five feet apart. The guns were fired from a position about five feet in front of the first screen.

At least two shots were fired for each cartridge. If the measurements did not agree closely, additional shots were fired and the results were averaged.

The Remington 158-grain (gr) RNL cartridge was fired from two Smith and Wesson Model 12-2 revolvers and from a Colt Cobra weapon, all having 2-inch barrels. This was done to test the consistency of cartridge performances in different guns which had the same barrel lengths. The velocities differed by less than one standard deviation.

Western Super X .22 caliber LR cartridges with 40-gr RN Lubaloy bullets were fired from both a Colt and a Smith and Wesson revolver, both having 6-inch barrels. The velocities differed by one standard deviation.

The foregoing .22 caliber LR cartridges were fired from the following weapons to verify the dominance of barrel length over other differences between weapons: (a) a revolver with a 2-1/4-inch barrel, (b) an automatic with a 4-inch barrel, (c) a revolver with a 6-inch barrel, and (d) an automatic with an 8-inch barrel. When plotted against barrel lengths, the velocities fell on a straight line (curve No. 4 in Figure 6). The remaining curves in Figure 6 were plotted from data published in the August, 1976, issue of "Guns and Ammo." These curves show the effect of barrel length on the bullet velocities of two .38 Special and two .357 Magnum cartridges, all fired from a Dan Wesson revolver with interchangeable barrels.

^aManufactured by Oehler Research, Austin, Texas.



Figure 6. Velocity as a Function of Barrel Length

In general, cartridges that are highly loaded for the weight of the bullet show a linear increase in the bullet's muzzle velocity with increases in barrel lengths. The slope of the curve (Figure 6) diminishes when the longer barrel lengths are used with lightly loaded cartridges and for cartridges using fastburning powders. Very lightly loaded cartridges show insignificant increases in muzzle velocities when longer barrels are used.

Different batches of what is ostensibly the identical ammunition may differ in their characteristics. Three boxes of .38 special Remington 158 grain RNL cartridges, purchased over a period of 18 months, had 3.76, 3.85, and 4.21 gr of identical looking powder per cartridge, respectively, and had different velocities when fired. This is probably the principal reason for discrepancies in published velocities. Our own measurements were always performed on cartridges from the same box of ammunition that was used for the characterization studies. Manufacturers' specified velocities are usually measured by shooting from test barrels, most often 10-in. long, and are not representative of velocities from handguns.

B. Characterization of Residue.

1. Overview. To characterize the residue from a particular cartridge, it is essential to start with a clean gun, otherwise residue from previous firings may be mixed with residue from the current firing. On several occasions, antimony-containing particles were found in test-firings of .22 caliber ammunition whose primer did not contain antimony. The antimony was never in evidence if the gun was given a thorough cleaning prior to firing the antimony-free ammunition. Particles containing copper seem to have even more tenacious retention characteristics. If plated or jacketed bullets were previously shot from a weapon, it is common to find a few particles containing copper in residue from plain lead bullets. These are few in number compared with plated or jacketed ammunition which usually gives residue in which many more particles contain copper. Zinc may be observed to accompany copper in some particles. Some platings or jackets are yellow brass, containing 30 to 35 percent zinc, in which case most particles containing copper also contain zinc.

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Residues were characterized by firing a single round, one-handed, with clean hands. The firing hand was then sampled by a standardized procedure^a, and the process was repeated by either using the other hand of the same person or of another person. From three to seven rounds (five in most cases) were fired, involving from two to five different persons. The samples were then analyzed, and the results averaged. Standard deviations for particle counts ranged from 30 percent to 125 percent. The mean standard deviation was 76 percent and the standard deviation of the standard deviations was 30 percent. The deviations include the random variations from cartridge to cartridge, the differences between hands, variations in the pattern in which the sampling disk was moved about the hand, and the residual fluctuations due to extrapolation from the disk area actually surveyed to the total area of the disk. This extrapolation is discussed in VIII. C.

Standard deviations for the ratio of bullet to primer particles tended to be significantly lower than the standard deviations for total particle counts. For example, in one test series the mean of six determinations was 5315 ± 3622 (68 percent) for total particles, while the mean for the percentage of primer particles was 50.5 ± 14.4 (29 percent). In another test series, the mean of 5 determinations was 203 ± 81 (40 percent) for total particles and 65.4 ± 10 (15 percent) for the percentage of primer particles.

Assuming a standard deviation of 100 percent for particle counts, it follows that variations by factors of three of more from the mean are highly unlikely. With some exceptions, variations of this magnitude were not observed.

Particle counts 8 to 10 times the mean were observed in a few firings. This was tentatively attributed to deterioration of the smokeless powder or to a defective primer which caused less than full power to be developed, with a resulting lowered muzzle velocity and a higher particle

^aThe numbers of particles listed in Tables 3 through 6 apply when the standard sampling disk is used as described in this report. The use of other equipment or procedures will require the numbers to be multiplied by a normalization factor.

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count. Higher particle counts could also result from misalignment of the firing chamber and barrel, which would cause shaving of the bullet. Lower than expected particle counts can be attributed to a defective adhesive coating of the collection disk (resulting in a lower collection efficiency), or to faulty procedures.

2. Antimony. Particles containing antimony are defined to be particles in which antimony is a major or minor constituent. This does not apply to particles containing trace amounts of antimony, using the definitions given in IX. A of the terms major, minor, and trace. The designation of components as major or minor is based on x-ray intensities rather than on concentrations. Trace means "just detectable." The antimony content of bullet lead ranges from near zero to 3-1/2 percent, except when re-loaders use lead not specifically intended for bullet manufacture. When this occurs, the antimony content may exceed 4 percent. Shotgun pellets can range up to 6-1/2 percent.¹⁷

In the analysis of residue from some firings, trace amounts of antimony were found in many of the bullet particles. Presumably, such residue came from bullets with an antimony content near the high end of the quoted range. The detection limit for antimony in lead is poor because of the high absorption of lead for x-rays.

3. <u>Copper</u>. Copper is found in both bullet and primer particles if the bullet is plated or jacketed. The fraction of particles that contain copper and the division of that fraction among the various classes of particles has some degree of reproducibility for a given cartridge, but that reproducibility is poorer than is the case for the other characteristics measured. Furthermore, the variations from one cartridge type to another appear quite irregular, without any discernible systematic trends.

The occurrence of a few copper-bearing particles in residue from ammunition with bare lead bullets has been attributed in III. B. 1 to retention in the gun of residue from previous firings with plated or jacketed bullets. This retention is known to occur, as explained in the antimony discussion. There are other mechanisms by which copper can enter the residue, but these mechanisms can only play a subordinate role, as evidenced by the fact that if copper-bearing

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particles are found at all in residue from bare lead bullets, they are few in number. Quite rarely, microscopic fragments of brass are found in residue. These may have been torn from the front edge of the cartridge case. A quantity of brass may be vaporized from this edge and mix with the other residue vapors. Evidence for this occurrence is furnished by the infrequent observation of nickel in a residue particle which also contains copper and zinc. The nickel must come from the nickel plating of the brass case. It is also conceivable that copper could be evaporated from the anvil in the primer, but there is no evidence that this happens. Finally, as stated in II. A. 1, copper may be present in some primers, but this does not appear to be common, if it occurs at all.

4. Standard deviations. It is necessary to examine the meaning of a standard deviation that is a large fraction of the measured quantity, or which in some cases even exceeds it in magnitude. The following is a review of what a standard deviation is intended to convey. If repeated determinations of some physical measurement are performed, typically, the several determinations will result in slightly different numerical values. If one may assume that these differences are entirely due to random causes, the arithmetic mean (the average) of all the measurements is taken as a better approximation of the true value than any single one of the measurements. It is then possible to define a quantity, called a standard deviation, such that 2/3 of a large number of measurements will fall within a one standard deviation difference from the "true" value, 95 percent of the measurements will be within two standard deviations, and 99.7 percent will be within three standard deviations. Stating this somewhat differently, there is only a 0.3 percent probability that a single measurement will differ from the true value by more than three standard deviations.

Returning to the question posed at the beginning of this section, let us look at an example of a series of five measurements that exhibited greater than average variability. In cartridge Test No. 81, the following results were

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obtained for the count of spheroidal bullet particles that contained copper and are included in size range A:

Number (N) of Particles	10	log N
18	1.	.26
80	1.	90
13	1.	. 11
7	0.	85
7	0.	85

The arithmetic mean (\overline{N}) and the standard deviation (σ) of the number of particles are 25 ± 31. The standard deviation was conventionally calculated by:



If one adopts a 95 percent confidence level which corresponds to 2σ , the result means that 95 percent of the experimental data should range between -37 and +87. For those data that are higher than the mean, this estimate is both reasonable and in agreement with observations. It is not reasonable and not in agreement with those data that are lower than the mean. It is not meaningful to predict a lower limit of -37 when the lowest physically possible value is 0.

The experimental data for various cartridges are plotted on a logarithmic scale and establish the amount of residue to somewhat better than an order of magnitude. Typical variations range between one quarter and one half of an order of magnitude. The linear dimension along the scale for N is actually the logarithm of N. Therefore, it may make sense to investigate the variations in

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the logs of the numbers rather than the numbers themselves. The mean and the standard variation of the logs are:

$$log N = 1.19 \pm 0.43$$

Remembering that addition and subtraction of logs is equivalent to multiplication and division of the numbers, this converts to:

$$\overline{N} = 15.5 \begin{cases} x \\ \vdots \\ 2.70 \end{cases}$$

The new mean is on a non-linear scale and is not of particular interest. The new logarithmic standard deviation (designated by σ '), applied to the log mean as a multiplier and divisor, gives for 2σ ', a range of from 3 to 84 particles^a. This is not only reasonable, but is in excellent agreement with the data for both those above and below the mean. Analogous results were obtained for a few additional cases to which this procedure was applied.

This shows that the logarithmic standard deviation gives a more reasonable assessment of the precision of numbers that vary over an appreciable fraction of an order of magnitude and are recorded on logarithmic scales. Except for the somewhat more variable counts of copper-containing particles, the standard deviations found in this work, expressed as multiplicants/divisors, are factors that range from 1.5 to 3. Specific values are not quoted for individual tests, because the number of determinations involved, generally five, is too small a number for statistical theory to apply rigorously. Quoting specific values might therefore imply more confidence than is warranted.

Implicit in the treatment given in the foregoing discussion is the conclusion that the data for the numbers of particles in a test series have a lognormal rather than a normal distribution. This means that a plot of the

^a15.5 x 5.4 \approx 84

 $15.5/5.4 \approx 3$

logarithms rather than a plot of the numbers themselves has the shape of the standard (Gaussian) error curve, i.e., a normal distribution. There are not enough measurements to demonstrate this conclusively, but the logarithmic dependence on velocity and the reasonable fit of the logarithmic standard deviations $l \sim l \sim l$ suggest that it is true. Log-normal distribution statistics were also found by the Gulf General Atomic Group in a different treatment of their data.²

For the log-normal distribution, the relationship between σ ' and probability is somewhat different; 3σ ' is equivalent to a 93.3 percent probability and 4σ ' to 97.7 percent.

C. Identification of Tests.

Gun-cartridge combination test firing results are identified by two-digit or three-digit numbers in subsequent chapters of this report. All digits except the last digit of these numbers identify the gun as it is listed in Table 1 under the column entitled "Gun ID No." The last digit refers to the cartridge within a particular family as listed in Table 2 under the column entitled "Test ID Within Family." For example, 12 refers to a .38 caliber S&W revolver, Model 12-2, used with Federal 158-gr RNL ammunition.

D. Results by Cartridge.

1. <u>The .22 caliber LR cartridges</u>. Five different brands or versions of 40-grain round-nose ammunition were tested. One had Lubaloy-plated bullets and the others had plain-lead bullets. Two revolvers and one autopistol were used, all with different barrel lengths. Seven different combinations of the three guns and five cartridges were tested and the residues were characterized. The results are listed in Table 3. The relationship between the total number of spheroidal particles and the muzzle velocity of the bullet is shown by the upper solid line in Figure 7 on a semi-logarithmic plot. The least squares equation for this line is:

 $\log N = 9.59 - 0.00664 v$

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Gun				Nominal	Chambered	Barrel	Length
ID No.	Type	Make	Model	Caliber	for	in.	mm
		Hai	ndguns Used for Ch	naracteriza			
1	Rev	S&W ^a	12-2	. 38	SPb	2	51
2	Rev	H&R ^C	929	. 22	LR ^d	2-1/2	64
3	Auto	Llama		. 32	ACP ^e	3-1/2	89
4	Rev	S&W	10-4	. 38	SP	4	102
5	Auto	Browning	Std	.380	ACP	4-7/16	113
6	Auto	Browning	High-Power	9mm	Luger (Parabellum)	4-2/3	119
7	Auto	Ruger	Std	. 22	LR	4-3/4	121
. 8	Rev	Colt	Officers' Model	. 22	LR	6	152
9	Auto	Hi-Standard	M-101	.22	LR	6-3/4	171
10	Rev	S&W	27	.357	Magnum	6	152
11	Auto	Colt	"Automatic Calibre 25"	.25	ACP	2	51
		Additional Han	dguns Used for Vel	locity Meas	urements Only	7	
1A	Rev	S&W	12-2	. 38	SP	2	51
1B	Rev	Colt	Cobra	.38	SP	2	51
8A	Rev	S&W		.22	LR	6	152
12	Auto	Colt	1911	, 45	ACP	5	127

Table 1. List of Handguns

^aS&W = Smith and Wesson

^dLR = Long Rifle

^bSP = Special

^CH&R = Harrington and Richardson

^eACP = Automatic Colt Pistol

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		F	Bullet			Test ID							
Caliber and Family	• Mfr ^a	Wt (gr)	Type ^b	Series	Velocity (fps) For Barrel (in.)	Within Family	Weight	Type ^c	Size, mm				
.38 Special	REM	158	RNL		703(2), 758(4)	1	4.21 gr (.273g)	Disk	.8				
	FED	158	RNL		706(2), 712(4)	2	3.52 gr (.228g)	Disk	. 8				
	w-w	158	LHP	Super X	806(2), 863(4)	3	5.76 gr (.373g)	Disk	1.4				
	REM	125	SJHP	+P	875(2), 885(4), 950(6)	4	7.75 gr (.502g)	Disk	1.5				
	w-w	148	wc	Super Match	665(2 and 4)	5	2.40 gr (.156g)	Flake	1.0				
	REM	158	RN, FMJ	"Metal Point"	682(2), 767(4), 769(6)	6	3.80 gr (.246g)	Disk	. 8				
	CCI-Speer	148	HBWC		627(2), 604(4)	7	2,79 gr (.181g)	Disk	.8				
	SV	110	SJHP			8	7.88 gr (.511g)	Disk	1.5				
.357 Magnum	REM	125	SJHP	High Speed	1447(6)	1	19.81 gr (1.284g)	Flat Ball	. 2				
	NORMA	158	SPFN		1217(6)	2	14.12 gr (.915g)	Cy1	.4-1,0 x.4				
a -	W - W	158	SWC Lubaloy		1425(6)	3	14.67 gr (.951g)	Flat Ball	0,2				
	sv	110	JSP (SJFN)		1522(6)	4	9.83 gr (.637g)	Disk	1,5				

Table 2. Ammunition Used in Characterizations or Velocity Measurements

^aManufacturers:

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REM: Remington Federal

FED:

W-W: Winchester-Western

^bBullet Types:

- RNL: Round Nose, Lead LHP: Lead, Hollow Point SJHP: Semi-Jacketed Hollow-Point wc: Wadcutter
- RN: Round Nose

^cPowder Types:

Cyl: Cylindrical Flat Ball: Flattened Ball Powder Cyl:

CCI: Cascade Cartridge, Inc.

sv: Supervel

FMJ: Full Metal Jacket HBWC: Hollow-Base Wadcutter SPFN: Soft Point, Flat Nose SWC: Semi-Wadcutter JSP: Jacketed, Soft Point

A 111 1		I	Bullet			Test 1D	Powder						
Caliber and Family	Mfr ^a	Wt (gr)	Type ^b	Series	Velocity (fps) For Barrel (in.)	Within Family	Weight	Type	Size, mm				
.22 LR	W - W	40	RN Lubaloy	Super X	858(2-1/2), 1000 (4-3/4), 1066(6), 1128(6-3/4)	1	2.36 gr (.153g)	Ball	. 2				
	W - W	- 37	HP Lubaloy	Super X	857 (2-1/2)	2	2.22 gr (.144g)	Flat Ball	. 35				
	FED	40	RNL	Powerflite, High- Velocity	969(2-1/2), 1168(6)	3	1.92 gr (.124g)	Disk, Flake	.7				
	FED	40	RNL	Champion	815(2-1/2)	4	1.55 gr (.100g)	Disk, Flake	.7				
	REM	40	RNL	Pistol Match	848(2-1/2)	5	1.21 gr (.078g)	Disk	.9				
	CCI	40	RN, Copper- Plated			6	1.86 gr (.121g)	Disk	. 8				
.380 ACP ^C	sv	88	JHP		1044(4-7/15)	1	3.86 gr (.250g)	Disk	. 8				
1	w-w	94	RN, FMJ		837(4-7/16)	2	3.09 gr (.200g)	Flakes	.4-2.0				
	REM	95	RN, FMJ			3	3.23 gr (.209g)	Disk	. 8				
9mm	FED	123	RN, FMJ		1144(4-2/3)	1	5.20 gr (.337g)	Disk Cyl	. 8				
	Speer	100	SJHP		1195(4-2/3)	2	5.73 gr (.371g)	Disk	1,7				

CCI:

sv:

Table 2. Ammunition Used in Characterizations or Velocity Measurements (Continued)

^aManufacturers:

W-W: Winchester-Western

FED: Federal

REM: Remington

^bBullet Types:

RN: Round Nose Hollow-Point HP: RNL: Round Nose, Lead RN: Round Nose

Jacketed, Hollow-Point JHP: FMJ: Full Metal Jacket SJHP: Semi-Jacketed Hollow-Point

Cascade Cartridge, Inc.

Supervel

^CACP: Automatic Colt Pistol (this identifies short Auto-Pistol cartridge styles)

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Table 2. Ammunition Used in Characterizations or Velocity Measurements (Continued)

-		E	Bullet			Test ID	Powder					
Caliber and Family	Mfr ^a	Wt (Gr)	Typeb	Series	Velocity (fps) For Barrel (In.)	Within Family	Weight	Type	Size, mm			
.32 ACP ^C	Browning	71	RN, FMJ		868(3-1/2)	1	2.17 gr (.141g)	Disk	. 8			
.25 ACP ^C	W-W	50	RN, FMJ		31(2)	1	1.21 gr (.079g)	Disk	.8			
.45 ACP ^C	Norma	230	SJHP		798(5)	1	4.85 gr (.314g)	Cyl	.4-1.0x.4			
	REM	185	SWC	Target Master	725(5)	2	3,88 gr (.25ig)	Disk	. 8			
	W-W	230	RN, FMJ		805(5)	3	5.24 gr (.340g)	Flake	1.2			
	sv	190	SJHP		1017(5)	4	7.78 gr (.504g)	Disk	1.5			

^aManufacturers:

W-W: Winchester-Western REM: Remington

sv: Supervel

^b<u>Bullet Types</u>:

Round Nose Full Metal Jacket RN:

FMJ:

Semi-Jacketed Hollow-Point SJHP: SWC: Semi-Wadcutter

^CACP: Automatic Colt Pistol (this identifies short auto-pistol cartridge styles)

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		roidal ticles Fraction	Percentage of Irregular Particles	Velo	ocity	Туре	Barrel		llet	in P	t Part ercent Size F		in P	er Part ercenta Size Ra	ages	Percentages of Particles Containing Copper				
Test No,	No.	from Bullet	Above Spheroidal	fps	m/sec	of	Length (in.)	Weight (gr)	Type & Brand	A	В	с	A	в	с	Bullet	Primer	Irregular		
24	16, 800	. 97	0.4	815	248	Rev	2-1/2	40	Fed Champ RNL	94	ь	ь	39	56	Ъ	N/A	N/A	N/A		
25	17,700	. 99	1.9	848	258	Rev	2-1/2	40	Rem Pistol Match RNL	92	b	Ъ	9 9		ď	N/A	N/A	N/A		
21	3, 550	. 93	1.0	858	262	Rev	2-1/2	-40	W Sup X RN Lubaloy	91	ь	Ъ ;	36	58	ь	76	13	53		
Meanc	9,800	. 96	1.5	853	260	-	4													
23	2, 426	. 92	4.5	969	295	Rev	2-1/2	40	Fed Powfl RNL	85	Ъ	Ъ	46	44	Ъ	N/A	N/A	N/A		
71	346	. 88	7.2	·1,000	305	Auto	4-3/4	40	W Sup X RN Lubaloy	83	b	Ь	28	58	Ъ	38	2	12		
81	239	.80	20,5	1,066	325	Rev	6	40	W Sup X RN Lubaloy	44	53	ь	30	45	b.	44	19	49		
83	110	. 72	23.6	1,168	356	Rev	6	40	Fed Powfl RNL	48	42	Ь	10	23	48	N/A	N/A	N/A		

Table 3. The .22 Caliber LR Cartridge Test Results

^aRange A: 1/2 to 2 μ m Range B: 2 to 8 μ m Range C: 8 to 32 μ m

^bMinor

^CThe mean of two cartridges with nearly the same velocity (Test Nos. 25 and 21). NOTE:

Barrel Lengths

2-1/2 in, = 6.34 cm 4-3/4 in, = 12.07 cm 6 in. = 15.24 cm

Bullet Wt

 $40 \, \text{gr} = 2.59 \text{g}$



Figure 7. Number of Spheroidal Particles vs Muzzle Velocity

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where N is the total number of spheroidal particles on the sampling disk, and v is the muzzle velocity in ft/sec. N declines one order of magnitude for each 145 ft/sec increment in velocity.

Within the precision of the measurements, the decline is the same whether the velocity is increased by firing the same cartridge from increasingly longer barrels, as in the series, (21, 71, 81) or (23, 83), or whether it is increased by firing more and more powerful cartridges from the same gun, as in test series (24, 25, 23) or (81, 83). This equivalence is emphasized because it does not seem to hold for .38 caliber cartridges (see D.2).

Along with the drastic decline in the total number of spheroidal particles with increased velocity, goes a modest but definite change in the ratio of bullet to primer particles. The percentage of bullet particles decreases from 97 to 72 percent over the range covered by the tests. Over the same range, there is a substantial increase in the proportion of irregular particles. Expressed as an added percentage over and above the spheroidal particles which are taken as 100 percent in each case, the irregulars increase from near zero to over 23 percent. A further change with increasing velocity is a gradual increase in the average size of the particles. For example, the percentage of bullet particles in the smallest size category (size A) decreases from 94 to 48 percent, with a corresponding increase in the larger categories. The tabulation of these data in Table 3 shows the following striking exception to this rule: In test 25, 99 percent of the primer particles fell into Size Range A, well outside the trend of the remaining tests. The primer of this particular cartridge does not contain any barium compounds. There is reason to believe that the presence of barium favors the formation of larger particles. Particles in which barium is predominant tend to be among the largest observed.

2. <u>The .38 special cartridges, 9mm, and 9mm short (.380 ACP).</u> The .38 special tests involved six cartridges and two revolvers with different barrel lengths. Four of the cartridges had bare lead bullets of either roundnose, wadcutter, or hollow-point construction. One cartridge had a jacketed round-nose bullet (FMJ), and the last a semi-jacketed hollow-point. Altogether, nine gun-cartridge combinations were tested. The results are tabulated in Table 4.

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-		roidal ticles Fraction	Percentage of Irregular Particles	Vel	ocity	Type	Barrel		llet	in P	t Part ercent Size R	ages	Prime in Pe Per S	r Part ercente Size Ra	iges	Perce Co	ntages of ntaining (Particles Copper
Test No.	No.	from Bullet	Above Spheroidal	fps	m/sec	of	Length (in.)	Weight (gr)	Type & Brand	A	в	c	A	в	c	Bullet	Primer	Irregular
15	7, 600	. 39	-0	665	203	Rev	2	148	ww wc	90	ь	ь	41	59	ь	N/A	N/A	N/A
11	3,200	. 78	1	703	214	Rev	2	158	Rem RNL	97	ь	ь	45	50	ь	N/A	N/A	N/A
12	2,900	• 96	0	706	215	Rev	2	158	Fed RNL	31	Ъ	Ъ	96	ь	b	N/A	N/A	N/A
41	6, 500	. 82	1.6	758	231	Rev	- 4	158	Rem RNL	91	b	Ъ	64	30	b	N/A	N/A	N/A
13	100	. 93	7	806	246	Rev	2	158	WW Sup X LHP	42	45	b	0	71	29	N/A	N/A	N/A
16	100	. 45	42	682	208	Rev	2	158	Rem Metal Point FMJ	33	33	34	11	45	34	0	13	21
46	1, 400	. 73	40	767	234	Rev	4	158	Rem Metal Point FMJ	7*	26	b	16	57	24	1	. 2	4
14	75	. 35	8	875	267	Rev	2	125	Rem SJHP	48	40	b	12	57	29	5	0	33
44	200	. 32	21	885	270	Rev	4	125	Rem SJHP	53	45	ь	33	55	12	3	8	7

Table 4. The . 38 Special Cartridge Test Results

² Range A: 1/2 to 2 μ m Range B: 2 to 8 μ m Range C: 8 to 32 μ m

^bMinor

NOTE:

Barrel Length:

2 in. = 5.08 cm 4 in. = 10.16 cm

Bullet Wt: 125 gr = 8. 10 g

148 gr = 9.59 g

158 gr = 10.24 g

The decline in the number of spheroidal particles with increasing muzzle velocity is again apparent. The other trends that were quite regular for the .22 cartridges are much less regular, or absent. Instead, the distributions divide the cartridges into two distinct groups. With the exception of the wadcutter, the cartridges with bare lead bullets produce residues with a high percentage of bullet particles and a very low percentage of irregular particles, while the jacketed bullets (both full and semi) give residues that are much lower in bullet particles and much higher in irregular particles.

The similar behavior of fully jacketed and partly jacketed bullets can be understood by noting that the so-called "full metal jacket," while it covers the nose of the bullet, leaves its base exposed. The semi-jacket covers the base of the bullet, but (with some exceptions) it leaves part of the nose exposed, so that in both cases the lead of the bullet is partly covered and partly exposed.

Only a speculative explanation can be offered for the anomaly of the wadcutter's lower percentage of bullet particles. The wadcutter is unique in that its bullet is totally recessed into the case. There are some indications that most of the residue that is found on the hand may be produced early in the firing cycle. If most of the residue was produced before the bullet is entirely free of the case, the case could act like a part-jacket.

The tabulation of the data in Table 4 again shows one anomaly in the size distribution of the primer particles in test No. 12. This is a Federal cartridge, and its primer contains less barium than the primers used in most other . 38 caliber cartridges.

The many class differences between jacketed and non-jacketed . 38 caliber cartridges make it unlikely that both groups should be plotted on a single line to express the number of particles versus velocity relationships. In fact, when this was tried and a least-squares line was put through all the points, some of the points were far off the line. For this reason, the data were evaluated separately for the two groups (see Figure 7). The steeply sloped line applies to the lead bullets. The equation of the line i $\log N = 11.16 - .0108v$. The dotted line, whose equation is: $\log N = 3.15 - .00102v$, is the least squares

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extrapolation of the four tests for jacketed bullets. This dotted line was also a good fit for two tests of .380 and two tests of 9mm ammunition, all jacketed. Although these are different types of cartridges, their bullet diameters within the usual tolerances are the same as .38 special ammunitions, and many of the bullets themselves are interchangeable. Consequently, a new least-squares calculation was performed for the combined group of the four jacketed .38 caliber cartridges and the four others. This resulted in the solid line that differs very little from the dotted line in Figure 7. Its equation is:

 $\log N = 3.64 - .00162v.$

However, it must be noted that in contrast to the. 22 caliber cartridges, a four-inch barrel gave more residue on the hand than the same ammunition shot from a two-inch barrel, in spite of the higher velocity it produced. This difference was observed for all three of the pairs tested, namely (11, 41), (16, 46), and (14, 44), which are connected by arrows in Figure 7. This behavior suggests that the number of particles ejected onto the hand is not only inversely proportional to the bullet's muzzle velocity, but also has a weaker direct relationship to barrel length, possibly due to the stripping effect of the rifling on the bullet. In the case of the .22 caliber cartridges, longer barrels produced considerable increases in velocity, causing the velocity effect to predominate. For the .38 Specials, the increase in velocity was much more modest, allowing the direct effect of barrel length to be observed. The existence of these effects seems established, but the reasons for their existence ure not understood.

3. <u>The 9mm Parabellum (Luger) and .380 ACP (9mm short) cartridges</u>. Only two types each of the 9mm and .380 ACP (9mm short) cartridges were tested. Some of the regular trends noted for the .22 caliber cartridges are apparent in this ammunition as shown in Table 5. Test 51 requires additional comment, because the percentage of spheroidal bullet particles has dropped to zero. The jacket of this bullet resembles the semi-jackets found on most

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		eroidal rticles	Percentage of Irregular						rcent	ages		rcenta	iges		ntages of ntaining	Particles Copper		
Test No.	No.	Fraction from Bullet	Particles Above Spheroidal	fps	m/sec	of	Barrel Length (in.)	gth Weight Type &		Per Size Range ²		Per Size Range ^a			Bullet Primer Irregu			
· · · · · · · · · · · · · · · · · · ·	- b		L <u>.</u>		9	l Imm F	arabellu		ger) Cart	ridges	L	L	L	Į.,	1	I		
61	166	. 40	4.8 ^b	1, 144	349	Auto	4-2/3	123	Fed FMJ	3	42	51	1 -	25	50 ^c	3	3	50 ^d
62	26	. 19	57.7°	1, 195	364	Auto	4-2/3	100	Speer SJHP	0	60	40	0	29	62	0	0	23 ^d
			••••••••••••••••••••••••••••••••••••••			380	ACP (9	mm Sh	ort Carts	idges)	<u></u>	•	.	A <u></u>	4			
53	244	, 17	22.6 ^f	840	256	Auto	4-7/16	95	Rem FMJ	73	27	0	3,5	51	34	0	4	2
51	52	0	23 ^h	1,044	318	Auto	4-7/16	88	sv ⁱ JHP	N/A	N/A	N/A	0	21	77	N/A	12	25

Table 5. The 9mm Parabellum (Luger) and .380 ACP (9mm Short) Test Results

^aRange A: 1/2 to 2 µm Range B: 2 to 8 µm Range C: 8 to 32 µm Ranged : 732 µm ^b1.2 percent Irregular 3.6 percent Clusters ^dIrregular only, no Copper in Clusters ^f21 percent Irregular 1.6 percent Clusters h21 percent Irregular 2 percent Clusters

^cSize D = 24 percent

^e50 percent Irregular 7.7 percent Clusters ^gSize D = 12 percent

Bullet totally enclosed

NOTE

Barrel Length:

4-2/3 in. = 11.85 cm 4-7/16 in. = 11.27 cm

Bullet Wt.:

88 gr = 5.70g 95 gr = 6.16g 100 gr = 6.48g 123 gr = 7.97g

other hollow-points in that it covers the base of the bullet but, unlike other hollow-points, it rises along the sides of the bullet to the edge of the hole in the nose. This is called a totally enclosed bullet, and the formation of bullet particles would be expected to be suppressed more severely than in the case of a jacket that leaves some of the lead exposed. Despite the consistency of this explanation, it was not valid in the testing of another totally enclosed bullet in a one-round firing. A Winchester-Western 9mm Luger cartridge with a 100 gr JHP bullet gave 30 percent spheroidal lead particles. As stated in II.A, a small but unknown fraction of the bullet particles may come from the primer. Therefore, the effect of total enclosure needs further investigation. On the whole, the fraction of bullet particles seems to correlate with the area of exposed lead in the bullet.

4. <u>The .357 Magnum cartridges</u>. A single revolver with a six-inch barrel was used to test three .357 caliber cartridges with jacketed bullets and one cartridge with a Lub loy-plated bullet.

The .357 caliber cartridge is approximately the same caliber as the .38 special or 9mm cartridges but, unlike them, it is combined with a magnum primer. Three points are not sufficient to define the correct line with confidence, especially since several of these tests had high standard deviations. The ammunition had been stored for some time. Nevertheless, the three points (not shown in Figure 7) are not inconsistent with a line that has the same slope as that for the jacketed .38 caliber cartridges. The displacement of the line towards larger numbers of particles can be attributed to the longer barrel and possibly the magnum primer. The point for the one plated bullet tested lies well above the others, again suggesting separate curves for jacketed and non-jacketed bullets. For the jacketed bullets, the percentages of bullet particles are high, but the jackets are relatively short and leave a substantial amount of lead exposed. The effect of velocity on the particle size distributions is evident in Table 6.

5. <u>Other caliber ammunition</u>. Only one other cartridge (.32 ACP) was characterized, and information on several more was obtained from test firings that were submitted by law enforcement agencies along with case evidence. The

Table 6. The .357 Magnum Cartridges Test Results

		eroidal ticles	Percentage of Irregular	Vel	ocity			Bu	llet	in Pe	t Parti ercent ize Ra	ages		er Part	ges		ntages of ntaining (Particles Copper
Test No.	No.	Fraction from Bullet	Particles Above Spheroidal	fps	m/sec	Type of Gun	Barrel Length (in.)	Weight (gr)	Type & Brand	A	в	C	A	В	С	Bullet	Primer	Irregular
103	6,650	. \$5	4	1,425	434	Rev	6	158	WW Sup X SWC Lubaloy	92	ь	Ъ	71	21	b	96	83	96
102	252	. 72	18	1,217	371	Rev	6	158	Norma SPFN (SJ SWC)	93	Ъ	Ъ	46	32	14	62	48	28
101	383	. 84	15	1, 447	441	Rev	6	125	Rem High Speed SJHP	96	b	b	39	23	32	74	29	55
104	225	. 89	11	1, 522	464	Rev	6	110	SV JSP (SJFN)	79	20	Ъ	17	67	Ъ	58	38	38

^aRange A: 1/2 to 2 μm Range B: 2 to 8 μm Range C: 8 to 32 μm ^bMinor

1

NOTE

Barrel Length:

6 in. = 15.24 cm

Bullet Wt.:

110 gr = 7.13g 125 gr = 8.10g 159 gr = 10.24g case test firings must be approached with some caution for characterization purposes, because the data were taken under less uniform circumstances, with weapons in unknown condition and with ammunition of unknown age. The greatest value of these data is for the cases from which they were taken.

In the tests performed at The Aerospace Corporation, only handguns ranging in condition from fair to new were used, and the only important parameters appeared to be the length of the barrel (regardless of make or model, or whether it was a revolver or semi-automatic. Derringers and guns of unusual construction were not tested). It is reasonable to expect, however, that a gun in poor condition may not give the same results. The condition of the barrel and the alignment of the firing chamber with the barrel could have an appreciable influence on the production of bullet particles. Abnormal clearances at the cylinder or breach end would affect the number of particles that can escape and the velocity with which they are ejected. The age of the ammunition can have a major effect on its overall performance. It is well known that ammunition can deteriorate. Police departments tend to consider ammunition unreliable if it is from six months to a year old. On the other hand, there are many examples of functional ammunition that is several decades old. One may speculate that a major factor that determines this difference, but which cannot be ascertained by inspection, is how gas-tight the seal is between bullet and case or between primer and case.

The .25 ACP, .32 ACP, .32 S&W, and .45 ACP cartridges fall reasonably close to the appropriate (jacketed or non-jacketed) .38 special cartridge curves (see Figure 7) at the indicated velocities and barrel lengths in total numbers of particles. The smaller calibers are on the low side in their fraction of bullet particles and on the high side in average particle size, compared to the .38 special cartridges. Judging from a very small number of cases, the .44 magnum ammunition gives extremely large numbers of particles, larger than that of any other cartridge residue examined.

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E. Origin of Hand Deposits

There is evidence that gunshot residue found on a shooter's hand is blasted onto the hand during the firing. Residue settling from the air does not seem to be a factor. If a bystander holds his own hand close to the side of a discharging gun, he can feel the impact of the particles, sometimes painfully. With some powerful cartridges, impacts were felt on the shooter's forehead, and the subsequent presence of residue on his forehead was confirmed by particle analysis. The settling time of small particles from the air is not fast enough (even for particles as heavy as lead^a) to account for the large numbers of particles acquired during the brief interval in which the hand is held out for firing. Furthermore, a settling mechanism would markedly favor an excess of larger particles, which is contrary to observed results. Although the evidence thus strongly supports the blasting mechanism, it does not explain differences between the indoor and outdoor firings observed in some previous work⁵ in which quantities of lead and antimony were measured by photoluminescence. The Gulf-General Atomic Group² failed to detect any influence of wind velocity on the quantity of residue.

The hand deposits are mainly breach deposits. They issue from openings around the breach mechanism and ejection port of automatics and the flash gap between the cylinder and the barrel of a revolver. Since in most automatics the location of the ejection port is asymmetric, differences between right-hand and left-hand firings were sought, but none were discovered. The differences were random and within the sample-to-sample variations.

Copious amounts of residue also issue from the muzzle, but appear to play a secondary role in the production of hand deposits. The hand deposits are therefore called breach deposits. Unlike the breach deposits, muzzle deposits which issue mostly in the forward (firing) direction, have not been systematically studied; however, some chance observations indicate that their particle distributions can differ from the breach deposits. Specifically, these observations

^aW.B. Renfro, thesis, Penn State University, June, 1972.

suggest that the muzzle deposits are richer in large flakes of smokeless powder and much richer in bullet particles, especially small lead spheres. There may be fewer irregular and fewer primer particles. If a hand deposit is found to depart considerably from what was expected on the basis of a breach deposit characterization study, and if the departures are in the directions indicated, it may be suspected that the hand deposit came from the muzzle blast rather than from the firing of the gun. These differences will be readily apparent only for those cartridges whose breach deposits are normally rich in irregular and primer particles.



CHAPTER IV. PERSISTENCE AND TRANSFER OF RESIDUE

A. Loss of Residue from Hands with Time.

The rapid decline of gunshot residue on the hand with time was noted in Chapter I, where Figure 1 illustrated both the decline of the bulk element quantities and the failure of many test firings to yield quantities that exceeded the significance levels or thresholds even when the residue was collected promptly. Particle analysis does not depend on a quantitative measure and can be successful as long as particles are detectable. However, since the number of particles does diminish with time, the rate of decline should be measured in order to determine how much time can be allowed to elapse between firing and collection before a search for particles may be fruitless. To this end, the following tests were performed:

> Each of five persons fired one identical cartridge. One person was sampled promptly and the others were sampled after delays of one, two, four, and eight hours, respectively. During the delay time, the persons involved pursued their normal activities in The Aerospace Corporation Laboratories, except that they did not wash their hands. This procedure was repeated four more times, rotating the participants among the various delay times. The results were averaged for each specific time period and plotted in Figure 8.

The decline in antimony levels from the .32 caliber ACP cartridge, as measured by photoluminescence, is shown again by the curve marked by solid triangles, and is compared with the decline in particle counts.

The top-most curve, marked with open squares, illustrates the decay of the total number of spheroidal particles in residue from a .38 caliber special cartridge. The bottom-most curve (solid circles) applies only to those particles that contained antimony in the same test. The second curve,

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Figure 8. Decline of Gunshot Residue on a Hand

indicated by open circles, applies to the total spheroidal particles from a .22 caliber cartridge. Within the precision of the measurements, all curves are approximately parallel and can be described by the simple equation which is plotted as a double line in Figure 8:

$$N_t = \frac{N_o}{10t}$$
,

where,

 N_t is the number of particles or the concentration after t hours N_o is the initial number or concentration

t is the number of hours elapsed between firing and sampling The test results show that:

- Particles were detectable after eight hours,
- The rate of decline is the same for particles as for bulk elements,
- The rate of decline is the same for antimony-containing particles (a small fraction of the total) as it is for the total of all particles,
- The rate of decline is the same for all calibers, and
- The rate can be represented by a linear equation. Numbers of particles have been plotted on a logarithmic scale in Figure 8 because they vary over several orders of magnitude, but time has been plotted on a linear scale because anything else would not be convenient. However, if both ordinate and abcissa were either logarithmic or linear, the plot would be a straight line.

The residue versus time relationship has been tested only for the first eight hours, after which it becomes academic. Hand-washing is more and more likely to occur with the passage of time. Also, the term "normal activity" is quite elastic. Some activities, especially those involving vigorous use of the hands, may remove residue at a faster rate than was experienced by our participants. Nevertheless, order-of-magnitude estimates based upon this equation have often been found to be applicable to actual cases.

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Persons at rest, and especially totally immobilized subjects (e.g., suicide victims if undisturbed) have a much lower rate of loss of residue. In two cases in which the hands of a suicide victim were strictly undisturbed, it has been possible to detect residue 3 and 5 days after the shot was fired.

B. Loss from Fabrics.

It was thought that woven fabrics might provide a better anchor for particles than skin, and that gunshot residue would therefore have a tendency to persist longer on clothing than on the hands.

Limited persistence tests were performed on a man's soft cottonpolyester shirt sleeve which was smooth and finely woven, and also on a laboratory coat that was more coarsely woven of a harder fabric consisting of 65 percent polyester and 35 percent cotton. The numbers of particles found in these tests after two hours differed by less than factors of three from those expected on the basis of the hand data. Since persistence inherently has large statistical variations, departures from expectations by factors less than four cannot be considered significant. Thus, it has not been established that the persistence of gunshot residue on fabric is different from that on hands. Therefore, sampling a suspect's sleeve may not be effective in extending the time limit within which gunshot residue can be detected. It would still provide a means of overcoming the effect of the suspect's having washed his hands prior to sampling.

With fluffy and deep-pile fabrics, sampling difficulties from excessive fiber pick-up were encountered (see X.A.). Also, it is doubtful that the flat layer of adhesive on the sampling disk can effectively reach many particles buried in the interstices of such a fabric.

C. Transfer of Residue.

Our case work indicates that guns which have been fired repeatedly and not cleaned are covered with residue that can be transferred to the web area of the hands of a person who handles that gun subsequently. While such

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deposits would be received initially in the palm of the hand rather than the back of the hand, it must be inferred that any kind of activity will quickly spread some particles around the hand. A moderately heavy particle deposit was also found on the back of the hand of a person who shook hands with another immediately after the latter had fired a gun.

Individuals standing abreast of, and in a line parallel with, the shooter were immediately sampled after a test firing. Those who were three feet away were found to have gunshot residue on their hands, but none was found on the hands of those who had stood ten feet away. These distances may vary with circumstances and the guns and ammunition used. Occasionally, clouds were seen to travel away from the gun almost at right angles, which is an observation also reported by others.¹⁹ An individual standing three feet directly behind the shooter was found to have only exceedingly sparse residue deposits on both his hands. In all these bystander tests, the bystanders had both hands at their sides. A short barrelled .22 caliber revolver was used (Test Combination No. 24).



CHAPTER V. OCCUPATIONAL AND ENVIRONMENTAL PARTICLES

A. Statement of Problem and Critical Occupations.

During the early phases of the development of particle analysis, gunshot residue was identified in each of the many hundreds of test firing samples collected. In many cases, the non-firing hand was sampled as a control, or hand-blank samples were taken from persons who had not fired a gun. No false positives were obtained from non-firing samples. Nevertheless, it was felt that better proof of the uniqueness of gunshot residue was required, and that an investigation should be made of the possibility that particles in the natural environment or particles produced by man could be mistaken for gunshot residue.

Small spheroidal particles of biological origin, such as pollen, cells, etc., are easily recognized in the SEM as being composed of organic materials and do not constitute a problem. Small spheroidal particles of an inorganic nature can be produced by milling, and by rapid condensation from a melt, and especially a vapor. Spheroidal particles containing elements heavier than sodium are not abundant. Irregular particles are the rule. Particles which give visible evidence of crystallinity can be immediately eliminated from further consideration. Particles of any description that contain combinations of lead, barium, and antimony are comparatively rare.

Industrial and commercial operations that involve the metals or compounds of lead, barium, and antimony were surveyed with special emphasis on activities that may involve melting and vaporization. Approximately 80 hand-samples were submitted for particle analyses. These samples were obtained from persons engaged in several critical activities and some marginally related activities. Many of the hand-samples, which included those considered most critical (lead smelting and explosive rivets), were submitted for analyses in blind tests (without identification), and were randomly intermixed with genuine gunshot residue samples. None of the occupational samples were falsely identified as gunshot residue by the experienced analysts; however, less experienced personnel might have encountered difficulties.

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B. Results of the Study of Occupational and Environmental Particles.

The results of investigations of particles in the environment or particles produced by various occupations are described in the following paragraphs.

1. <u>Studguns.</u> A studgun is a firearm used in construction for driving nails, rivets, and staples into hard materials, such as concrete, steel, and also wood. The cartridges used in studguns are comprised of two parts, which are a projectile and a "powercap" (Omark) or "powerload" (Remington). The projectile, which takes the place of the bullet, is a nail, rivet, or staple that is placed ahead of the powercap or powerload. The powercap or powerload can be made with "wad"-type construction. These have the appearance of blank cartridges, including the head stamps. They can also be made with "crimp"-type construction in which the front end is crimped to a fluted cone. The powercaps or powerloads are available in .22, .25, and .32 caliber sizes, with .22 caliber being the most common. Each caliber is available in several power or load levels.

Duplicate test firings were made of one. 22 caliber Remington wad type standard velocity (22W3) cartridge and one .22 caliber Omark crimp type low velocity cartridge. The studs fired were made of zinc-coated steel. The samples were analyzed in blind tests in which the operators had no information about the nature of the samples and performed a standard examination for the presence of gunshot residue.

When examining the first sample of the Remington cartridge, the operator performed an x-ray analysis for 40 particles and immediately eliminated 36 which contained no lead, barium, or antimony. Only 13 of the 40 were of spheroidal shape. The following are the characteristics of the four remaining particles:

Shape/Size (µm)	Major Elements	Minor	Trace
Irregular 2.5	Ba, Pb, Zn	Si, Fe	
Irregular 3.8	Ba, Zn, Si, Pb		Fe
Spheroidal 5.8	Ba, Si, Ca	(S), Al, Fe, Cl, Zn, Pb	
Spheroidal 2.9	Ca, Pb	Ba, Zn, Si, Fe, Cl, Cu	

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The first three were rejected because they contained zinc in the absence of detectable copper. The last one was rejected because the ratio of the zinc to copper peak heights was 5:1, the opposite of what might be given by a brass particle. The operator concluded that there were no particles consistent with gunshot residue.

The second test sample of the same cartridge (again analyzed blind) revealed 28 particles that contained Ba and Pb. All but one also contained Fe, and all but three contained Zn, including the one without Fe. Six of the Ba, Pb, and Zn particles were spheroidal, and the remainder were irregular. Many contained traces of Cu, but in only two of these were the copper peaks larger than the zinc peaks. Taken alone, these two are consistent with gunshot residue. A few of the Ba, Pb, and Zn particles also contained Mn and Cr, elements that, like Zn without Cu, are inconsistent with gunshot residue.

The two particles that were consistent with gunshot residue were questioned on the ground that they were associated with a larger number of very similar particles that were not considered to be gunshot residue. However, if these two particles had been the only ones found, they would have been accepted as being consistent with gunshot residue.^a

One of the Omark cartridge samples in the blind tests was dismissed by the operator as containing nothing of interest. Thirty-nine particles of possible interest were recorded for the second Omark sample, 12 of them spheroidal. They generally contained Ba and Pb and often Cu and Zn either as minor constituents or as traces. Thirty-five contained Fe. There were 13 particles in which the copper peak was higher than the zinc peak. A typical composition, with a 3:1 ratio for Cu:Zn, consistent with yellow brass, was

^aThe record of this examination also shows a few particles of Au, Ag, and Cu. This suggests jewelry. A ring worn by the carpenter who fired the studgun may have received some rough treatment. This is an indication of the high probability of detecting whatever is present, and that what is present may be unexpected and irrelevant. Pb, Fe, and Ba (major); Si, K, Cu, Ca, and Zn (minor). Such particles are consistent with gunshot residue. There were, however, many particles clearly inconsistent with gunshot residue, such as the following:

Maj	or				M	inor			
C1, S,	Zn	*	Cu,	Si,	Fe,	Ca,	К,	Ba,	$\mathbf{P}\mathbf{b}$
Ti, Ba	, Pb		Cu,	Zn					

In addition, there were two irregular lead-only particles, but no spheroidal particles. This is significant, because all but one of the pistol cartridges examined yielded spheroidal lead-only or lead plus copper particles, often in great excess.

The crimp-type powercap had a high incidence of Cu and Zn in proportions consistent with brass. This construction greatly increases the exposure of the front end of the brass case to the burning powder and could cause evaporation of copper and zinc.

The operators were impressed with the low percentage of spheroidal particles, the great sparsity of any lead-only particles, and the absence of spheroidal lead-only particles. They also noticed the presence of many particles similar to, but inconsistent with gunshot residue either because the particles contained elements not found in gunshot residue, or because the Cu:Zn ratio was wrong. Finally, it seemed that the percentage of particles that were similar to gunshot residue particles, but which contained Fe, was too high for any except steel-jacketed bullets.

Considering the basic similarity of studguns and conventional firearms, it is understandable that the operators, who had not previously seen residue from studguns and had not known the nature of the samples, were cautious in their verdict. Noting the differences enumerated, they refused to call the samples gunshot residue despite the presence of some particles consistent with gunshot residue.

The rule that emerges (which has already been given in Chapter II) and which will exclude studgun residues, is that the presence of substantial numbers of inconsistent particles overrules the evidentiary significance of particles consistent with gunshot residue. If the sample had been a mixture of genuine gunshot residue and studgun residue, the rule would result in a false negative, but it would prevent the possibility of a false positive.

2. <u>Children's cap guns and blank pistol cartridges.</u> Residue from children's cap guns contains antimony but no lead or barium. Distinctly crystalline fragments of an antimony compound were much in evidence, as were others which contained S\$, S, and K as major and Cl and P as minor constituents. There were particles, some spheroidal, that contained K, Cl, Ca, P, and others that had Ca, P, Zn, K as major and Sb, Cl, S as minor ingredients. One spheroidal particle was found which gave only a phosphorus spectrum.

Two blank pistol cartridges we're examined. One was a Winchester . 22 short with black powder and the other a Winchester-Western . 38 special with smokeless powder. Neither of them gave any detectable gunshot residue or particles resembling gunshot residue, when fired from a clean gun.

3. Lead smelting. Hand samples were examined from 13 different persons performing various jobs in a lead smelting operation.^a While there were some minor individual differences, the samples were similar enough to permit the following summary:

Despite the wearing of work gloves, all workers sampled had numerous particles containing heavy metals on their hands. Twenty-five to 75 percent of the particles were spheroidal, with an average of 50 percent. The sizes fell mostly into categories A and B, with 1-2 μ m very common. In order of typical frequency of occurrence, the compositions were as follows: (1) Pb, Zn These constituted 12-90 percent of the total, usually they were the most

^aObtained from a lead smelting plant in Idaho by Mr. John F. Anderson, Director

numerous category

of the Eastern Washington State Crime Laboratory.

(2) Pb only

About equal numbers of spheroidal and irregular particles

- (3) Pb, Zn, Sb
- (4) Pb, Sb
- (5) Zn only
- (6) Some of the above with one or several of the following:

Cd, As, Ti, Mn, Te, Ag, Cr, Cu

Although compositions (2) and (4) are consistent with gunshot residue, the average 50 percent irregular morphology is not consistent, and neither are the remaining compositions in the list.

The following is a list of the occupations of the subjects along with special features of the residues.

Subject Occupation	Special Features of Residue
Lead Refinery Operator	Pb-only particles most common
Blast Furnace Topper	No Cu containing particles
Blast Furnace Operator Foreman	All particles spheroidal. Most were
	Pb, Sb, Zn, most of remainder Pb,
	Zn
Fuming Plant Operator	Many Zn-only, no Pb-only particles
Cupola Operator	Typical, as described
Kettle Operator	Typical, as described
Laborer in Ore Mixing Area	Typical, as described
Sulfur Operator	Typical, as described
Security Manager	Typical, as described
Maintenance Man	Typical, as described
Pellet Plant Operator	Typical, as described, except very
	few spheroidal
Straight Line Machine Operator	Typical, as described, except very
	few spheroidal
Electrician	Typical, as described, except for one
	particle

The one non-typical particle found on the electrician's hand was a spheroidal particle, of 5 μ m diameter, and composition: Ba, Pb, Zn, Cu, Si, K, Ca, Fe. It was the only barium-containing particle in the entire lot.

As in the case of the studguns, all samples in this series were analyzed in blind tests. The operators' verdicts for all of these tests were "not gunshot residue." Possible difficulties can arise if very few particles are found, and the same comments made for studgun residues apply here.

4. <u>Automobile brake mechanic</u>. The samples described below were taken from the palms and backs of both hands of a subject who was employed in an automobile supply store and who turns brake drums.^a

Many irregular particles containing lead were found. Most of them contained one or several of the additional elements: Fe, Zn, P, Br, Cl, and Cr. The combination of lead with bromine is highly characteristic of the exhaust of automobiles running on leaded gasoline. Presumably, the turning of the drums produced steel particles containing Fe and Cr. The origin of the zinc is uncertain, but zinc is found in many parts of an automobile. Phosphorus is present in some gasoline and oil additives. There were nine barium-containing particles, which are listed here individually:

Particle Shape	Major Elements	Minor Elements
Spheroidal	Fe, Cu, S, Si, Ba, Pb	
	Pb, P, Ca	Ba, Zn, Fe
	Pb, Zn	Ca, Ba, Fe
Irregular	Ba, Pb, Mg	
	Ba, Pb, Mg, Cr	
	Pb, Ba, Zn, Fe	
	Ba, Ca, P, Pb, Zn	Fe
1	Pb, Fe	P, Ba, Zn, Ca
	Pb, Fe, Cu, Si, Ba	C1

^aThese samples and those from the battery assemblers tests (V.B.4) were provided by Mr. Gary Gonzales of the Orange County (CA) Sheriff's Crime Laboratory.

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Only the first of the spheroidal and the last of the irregular particles pass the compositional criteria for particles consistent with gunshot residue, but only marginally. There is a combination of lead and barium, accompanied by elements that can occur in gunshot residue. However, these additional elements are present in rather larger proportions than is typical for gunshot residue of this composition, so that these particles would be considered somewhat questionable even if found in isolation. Their association with other quite similar particles that do not meet the criteria would tend to rule them out, as in the similar circumstances of the residue from studguns.

Compounds of barium have wide industrial uses, including lubricating oil additives, greases, and thickeners for pastes of various kinds, pigment fillers and extenders for paints and rubber. Automotive brakes collect a miscellany of ground-up metal and brake lining particles, oil, grease, dirt and debris of various kinds. Turning brake drums is a good way of producing particles compounded of many such ingredients, which could explain the presence of barium.

5. Lead-acid battery assemblers. Samples were obtained from one person each in two different battery manufacturing establishments. The subjects were involved in assembling the battery plates. The traditional composition of such plates is an alloy of 94 percent lead and 6 percent antimony. $BaSO_A$ is used as a plate filler.

The samples from the first person engaged in this work contained many irregular and one spheroidal lead-only particle. In addition, the following three were of special interest:

Shape	Major	Minor
Spheroidal	РЪ	Sb
Spheroidal	Pb	Sb, Fe, Si, Zn
Spheroidal	Pb	Ca, Ba, Fe, Zn

The palm sample from the second person again contained many irregular Pb-only particles, but it also contained 40 spheroidal lead particles, many of which contained traces (of one or several) of Fe, Sb, Cu, Zn, Ba. The following three were typical:

Shape	Major	Minor	Trace
Spheroidal	Pb		Fe, Ba
Spheroidal	Pb, Cu, Si	Zn	
Irregular	Pb, Ca, Sb, 2	Zn, Si, Cu	

Of the spheroidal particles, one had a diameter of 2 and one of 3 μ m. The others were all 5 μ m or more in diameter, a size distribution not typical of most gunshot residues. With this many particles found, even those cartridges that give relatively large particles would have a few below 2 μ m. Some of these particles pass the compositional criteria for gunshot residue, and their probable true nature can only be inferred from the other particles with which they are associated.

6. Auto mechanics, automobile exhaust, environmental lead. The largest source of environmental lead contamination is leaded gasoline. Additional localized sources are lead smelting and secondary lead refining operations. Non-airborne lead contamination comes from peeling paint in old buildings. Lead issues from the exhaust of motor vehicles in the form of lead chlorides, bromides, oxychlorides and oxybromides. In moist air, these are slowly hydrolyzed to lead oxides, and some lead oxides issue directly from exhausts. However, most fresh exhaust that has been tested contains particles in which both lead and bromine are present. Also, the majority of casual lead particles found on the hands of people, those not due to gunshot residue or specific occupations, contain bromine and often chlorine. Chlorine is too ubiquitous to be an accurate indicator, but the presence of bromine in a lead particle can be taken to identify the latter as being from automobile exhaust. A minority of exhaust particles gives only a lead spectrum. The vast majority of automobile exhaust particles, with or without bromine, are irregular. A solitary spheroid is seen only occasionally. Spheroidal exhaust particles without bromine have not been observed in any of these tests.

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Automobile exhaust particles are abundant on the hands of many automobile mechanics that were sampled. They are found on the hands of service men who drive trucks from job to job, and they can be found occasionally on almost anybody's hand. The auto mechanics also had various kinds of metalcontaining particles, but none that were of interest in this study, except for the brake installer's already discussed.

7. Other occupations. None of the remaining hand-blanks from a large variety of occupations indicated potential conflicts with gunshot residue determinations; therefore, only some of them are briefly described.

The plumbers, machinists, laboratory technicians and, to a lesser extent, electricians, tended to have collections of mostly irregular particles with complex compositions on their hands. The compositions included a wide variety of common and sometimes uncommon metals, but no combinations other than lead particles that would meet the criteria for gunshot residue. Lead particles were also quite numerous on the hands of a glassblower, in addition to the same variety of particles already described.

The particles found on painters' hands are also of the complex, many-element variety, except that the quantity of lead particles is low. In recent years, lead has been almost eliminated from ordinary paints. It is present, however, in the oil paints of artists and in ceramic glazes.

Lead-tin combinations, which are indicative of solder, are found on the hands of television repairmen and electronic technicians. Spheroidal particles, which have the appearance of typical gunshot residue particles but which consist of iron, or combinations of iron and various alloying elements used in steel, are numerous on the hands of welders and flame cutters. They are tiny weld beads and are picked up occasionally by nearly everyone who handles objects made of iron or steel.

Another surprisingly common particle that looks like gunshot residue comes from the sparking flints used in cigarette lighters or the lighters used for gas burners, acetylene torches, etc. They are spheres, sometimes porous like a sponge, and consist of cerium and iron, often with lanthanum.

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Occasionally, one is found on the hand of a non-smoker. Sparking flints are made of a pyrophoric alloy of 70 percent "mischmetal" and 30 percent iron. Mischmetal is a mixture of rare earth elements from which the less abundant and more valuable elements have been removed. What is left may be mostly cerium, or a mixture of cerium and lanthanum, sometimes with traces of other elements.

8. <u>Chemical notes of interest on sources not tested</u>. Pyrotechnic materials (rockets, firecrackers, etc.) use black powder and powdered metals. Barium compounds are used to produce a green light, but antimony is not used. Lead (together with selenium) occurs only in delays for blasting caps. Similar delays in blasting caps for explosives contain lead, selenium, and tellurium as well; barium is used in gaslers fuse powders.

Lead-base babbitts and cerro alloys are low melting alloys of lead and antimony, most with detectable amounts of tin, that are used in fusible links for sprinkler systems, as fillers in bearings for machinery, and as fillers in automobile body repair work. It would be appropriate to examine handsamples from users of these materials. Time constraints have prevented this information from being obtained for this report.

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CHAPTER VI. DEMONSTRATION-EXPLORATION PROJECT OF CASE ANALYSES FOR LAW ENFORCEMENT AGENCIES

A. Purposes of the Demonstration-Exploration Project.

Occasionally during the earlier phases of this project, a particular analysis for gunshot residue was performed at the request of a law enforcement agency. At the beginning of 1977, an expanded program of such case assistance was made a formal part of the project. This was considered to be one of the necessary steps to advance particle analysis to a "field ready" status. The case assistance program had the following objectives:

- To detect and solve any remaining problems that may be encountered in actual cases.
- To speed the transfer of the method from the laboratory to field use.
- To acquaint more criminalists with the details of the method well enough to start them practicing it.

B. Types of Cases Handled and Success Rate.

Two months prior to the formal termination of the program, 100 cases had been completed, with eight more waiting in the queue and some additional cases expected. Ninety-three of the 100 completed cases involved samples taken from the hands of individuals, and 86 of these fall into the three categories tabulated in Table 7 which permits a straightforward evaluation.

The first category, labelled Homicides/Assaults, consists of cases in which someone fired a gun, but not at himself. Handsamples from one or several suspects, and in most cases a test-firing sample, were analyzed for the presence of gunshot residue. In order to be able to calculate a "success" rate, the assumption was made that at least one of the suspects was in fact the correct one, so that a successful analysis would consist of at least one positive handsample. Since this assumption could occasionally be wrong, equating positive results with successful analyses and negative analyses with failures ensure that the actual success rate is at least as high as the number

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		Rifle and	Rate of P	ositives
Category .	Handgun Cases (No.)	Shotgun Cases (No.)	Handguns (%)	Overall (%)
HOMICIDES/ASSAULTS:				:
Residue found on at least one suspect	28	5		
No residue found	2	4	88	79
Inconclusive	2	7		
SUICIDE/HOMICIDE DECISION:				
Residue on victim or suspect or both	8	-	89	89
No residue found	1	-		07
SUICIDE VERIFICATION:				
Residue on victim's hands	29	• 2		2.2
No residue found	2	2	94	89
OVERALL:				
Residue found	65	7	90	84
Not found or inconclusive	7	7		

Table 7. Summary of the First 86 Cases of Handsample Analyses^a

given, but it could be higher. Three cases are labelled Inconclusive. In each of these cases, only one or two irregular lead particles were found. This could have been gunshot residue, but was not characteristic enough to result in a positive finding. For purposes of statistics, these cases were combined with the failures, cases in which nothing was found that could be interpreted as gunshot residue.

^aFour individual case reports are shown in Appendix E.

On the foregoing basis, the success rate was 88 percent where handguns were involved and 50 percent for the much smaller number of cases involving long guns, for an overall rate of 79 percent. This work did not include a study of long guns, so that the conditions for detecting residue from long guns were not optimized. This category includes the most successful as well as the least promising case that was handled. One of the positive results for handguns involved a single shot from a .22 caliber revolver, with the suspect sampled 13 hours after the firing. He was asleep for 6 hours and awake for 7 of the 13 hours. This is the most successful case. One of the negative long gun cases involved a .22 caliber rifle. This was the least promising case because the suspect was sampled 15 hours after the shooting occurred. Generally, accepting cases for live suspects after more than a 12 hour delay is not recommended.

The second category, labelled Suicide/Homicide Decision, involved examination of samples from both the victim and one or more suspects. The assumption was again made that there should be at least one positive sample in the lot, other than the test-firing sample. This was found in eight of the nine cases, all of which involved handguns only, for a success rate of 89 percent.

The final category, labelled Suicide Verification, consists of cases for which samples from only the victim were submitted. For statistical purposes, it is assumed that all of these are bona fide suicides and require a positive result. This was obtained in 94 percent of the handgun cases and in 50 percent of the four cases involving long guns, for an overall rate of 89 percent. However, based on experience, a negative result in an apparent suicide by handgun seems highly suspicious and should be further investigated if it can be ascertained that the victim's body has not been disturbed up to the time of the sampling. The interpretation of evidence in apparent suicide cases is discussed in VI. D. 1.

Averaging all three categories, positive results were obtained in 90 percent of the handgun cases and 50 percent of the long gun cases, or 84 percent

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overall. Bearing in mind the assumptions made, these are the minimum numbers for the rates of success of particle analysis applied to 86 cases.

C. Impact.

The criminalists and detectives at whose requests the analyses were performed generally agree on the usefulness of the results in the investigative and pre-trial phases of cases. In a number of instances the existence of the evidence caused the defendant to change his plea. This includes a few lastminute pleas of guilty when Aerospace personnel had already been subpoenaed to give testimony.

Some cases are still pending. Information on the dispositions of the others is still being received and is currently incomplete. There were a number of convictions, but it is too early to assess the impact of particle analysis on the rate of convictions. Aerospace personnel have testified in court only twice. In one case, the defendant was convicted, and the testimony was material to the conviction. The other case was dismissed on technical grounds not connected with the analysis, although all testimony including that by Aerospace personnel was damaging to the defendant. A criminalist who had witnessed an analysis in the Aerospace Laboratories testified to it in court, and the defendant was convicted. Another criminalist testified at a preliminary hearing.

D. Special Problems.

1. Interpretation of the evidence in suicides. A person committing suicide obviously cannot hold the gun in the normal firing attitude which was used to arrive at the characterizations described in this report. Experience from cases suggests, however, that in most cases the number of particles deposited on the victim's hands at the time of firing is at least equal to that resulting from normal firing when a handgun is used. There is not enough experience with long guns to make a similar statement for these, but it would seem that they afford greater opportunities for variations because of the problem of manipulating the gun. The loss of residue from the hands of live subjects as a function of time probably consists in part of a simple falling off of particles, but loss may be accelerated by activities that tend to wipe the hands. Some suicide victims who died instantly and were sampled before the body was disturbed had ample residue on their hands as many as 5 days later, which was the longest delay encountered in any of the cases. Other cases tend to indicate that handling of the body and transportation can result in loss of residue to varying degrees. In the companion report on a field test of photoluminescence analyses for lead and antimony, some data are reported that show a correlation between the amounts of these elements found and the time lapse between death and sampling at the morgue.

There are indications that bagging of the hands may be detrimental rather than helpful in the preservation of the residue on the hand. The bagging practice is therefore viewed with reservations.

If the victim does not die immediately and is rushed to a hospital, the attempts to save his life obviously take precedence over preservation of the evidence, and the residue may be lost.

If it can be ascertained that the victim's hands were sampled before any serious disturbance of the body has taken place, a failure to find gunshot residue is suspicious enough to cause a further investigation into the possibility of a disguised homicide. However, if gunshot residue is found, this does not prove that the victim shot himself. The residue could have come from the muzzle blast of a gun held by another person. If the victim is aware of being attacked, especially at close range, he or she is quite likely to throw up his or her hands in a defensive gesture and thus receive the full muzzle blast on the hands. Sometimes it may be possible to distinguish between breach deposits and muzzle deposits, as described in II, E.

A difficult situation arises if both the victim and a potential suspect or a witness (who may also become a suspect) are found to have gunshot residue on their hands. If the suspect's residue is ample and the victim's sparse, homicide appears likely. In the reverse situation, the explanation may be accepted that the victim committed suicide, and that the survivor

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picked up some residue by handling the gun or by handling the victim to see if he was still alive or to assist. However, if a few hours have elapsed before the survivor is sampled, the small amounts of residue found on him may be consistent with his having fired the gun a few hours earlier. In the light of all the circumstances, it will then have to be decided whether or not the survivor is a suspect in a possible homicide.

2. <u>Samples contaminated with blood</u>. Samples were frequently received in suicide cases in which dried blood was present on the surfaces of the disks. This has never interfered with the detection of gunshot residue by particle analysis.

3. <u>Perspiration</u>. No difficulties were encountered with samples taken from heavily perspiring hands. It may be that residue clings better to moist hands and that this compensates for a possible lowering of the effectiveness of the adhesive.

4. <u>Time between firing and sampling</u>. The average time lapse between firing and sampling was 3-1/4 hours, with a range from one to 13 hours for live subjects. There was no difference in the average time lapse between cases with positive and negative results. Considering handguns only, the four negative results all involved cartridges that tend to give sparse residues, but these cartridges were also adequately represented among the positive results, so that this cannot be considered the determining factor. Two of these four cases took place indoors and two outdoors.

The victims of either suicide or homicide tended to be sampled much later. The average time lapse was 15 hours, with a range from 1-1/2 to 120 hours. The suicide victim that was sampled 120 hours later yielded ample residue, more than many others. Presumably, this body was sampled while still completely undisturbed.

5. Long guns. During the course of this program, residue from the firing of some rifles and shotguns was collected for characterization, but ultimately it was not possible to include this subtask within the scope of the program. Thus, the only information acquired about residue left on hands

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by long guns comes from the 15 cases submitted by law enforcement agencies that involved the use of such weapons.

It is hazardous to draw conclusions from such a small number of cases. Shotgun cases gave 4 positive results out of 5 and rifles only 3 out of 8. Of the three positive rifle examinations, two involved bolt-action guns, and one was a semi-automatic. The five negatives were either semiautomatic or lever-action rifles. It is still assumed that all results should have been positive. A .22 rifle used to fire birdshot was not included in this summary because no decision was reached as to whether it should be treated as a rifle or shotgun case. It gave a negative result.

It appears that rifles deposit much less residue on the hand than do handguns, and it may be that less residue escapes at the breach. Alternatively, it is possible that the spatial distribution pattern is different. No attempt was made to look for residue on the face of the shooter. This may be a worthwhile experiment.

E. Statistical Information.

Table 8 is a summary of the number of cases in which guns were encountered that were chambered for the cartridges listed. Table 9 is a breakdown by make of gun and the brand and style of the ammunition used. Some of this information is incomplete. It is listed exactly as reported, with no effort at consistency. For example, Win (Winchester) and W-W (Winchester-Western) refer to the same manufacturer, but the ammunition may have been marketed under different brand names, or it may be old ammunition produced before a merger, etc.

These tables include cases beyond those listed in Table 7. They were not included there because they did not fit the three standard classifications. Also included are cases not yet completed. Some cases involved more than one weapon.

F. Time Required for Analysis.

The time spent on analyzing a sample varies with the fraction of the area examined, the amount of gunshot residue found, and the amount of extraneous

Caliber	Type	Number of Times Encountered in Cases
.38 Caliber:		
. 38	Special	27
.357	Magnum	7 (Two were used with .38 Special ammunition)
. 38	S&W	2
.380	ACP	2
9mm		4
	Total of .3	8 caliber cases 42
.22 Caliber:		
. 22	Long Rifle	22
. 22	Short	4
. 22	Magnum	2
	Total of .2	22 caliber cases 28
.32 Caliber:		
. 32/20		1
. 32	S&W	8
. 32	ACP	2
-	Total of .3	32 caliber cases 11
Other Caliber	s, Rifles, a	and Shotguns:
. 25	ACP	8
. 44	Magnum	2
. 45	ACP	1
Unknown		3
Rifles		10
Shotguns	, · ·	7
	Total of ot	her calibers,
		nd shotguns 31
	Total of al	l weapons listed 112

Table 8. Weapon Statistical Information

	Gun			Ammunition			
Make	Model	Barrel Length (in.)	Brand	Bulle Type	Weight		
20.0		(111.)	l	l	(gr)		
.38 Special	· · · · · · · · · · · · · · · · · · ·				1 127		
S&W		6	R-P	SJHP	125		
S& W		6	Rem	RNL	158		
S&W		4	Speer	SJHL	125		
S&W	н. — — — — — — — — — — — — — — — — — — —	4	W-W	RNL (Super X)	1.		
S&W		4	W-W	RNL			
S& W	64-1	4	W-W	LHP+P	:		
S& W	Combat Masterpiece	4	sv	SJHP	110		
S& W	Chief	2	Reload	wc	148		
S&W		4	sv	SJHP	110		
S& W		5	R-P S&W	RNL SJHP	157 110		
5&W		4	w-w	RN Lubaloy	158		
5&W		4	Rem	FMJd	158		
S& W	10-5	2	Rem	RNL	158		
Colt	Cobra	2	Rem-UMC WRA	RN Lubaloy	158		
Colt	Agent	2	w-w	RNL (Reload ?)	~154		
Colt	I.	4	S&W	RNL	158		
Colt		2	Rem	SJHP	125		
Rohm	RG 38T	6	R-P	RNL	158		
Rohm	385	4	Reloaded Milit.	RNL (WCC 71)			
Charter Arms	Undercover	3	R-P	RNL	158		
Charter Arms	Undercover	2	W-W	RN Lubaloy	158		
INA		2	sv	SJHP	1		
H&R	926	4	R-P	RNL			
Taurus		3	Western	RNL	158		
Enfield		5	S&W	Lubaloy	145		
Unknown			Unknown	1			
Unknown			Unknown	(WC ?)			

Table 9. Weapon and Ammunition Makes and Models



	Gun			Ammunition	
		Barrel		Bullet	
Make	Model	Length (in.)	Brand	Туре	Weight (gr)
.38 Smith & Wesse	on			1	
Ivers-Johnson		5	WRA	RNL	145
Webley (type)	Standard, British Milit.	5	R-P	RNL	146
357 Magnum					
Colt	3		Reload	.38 Spec.	
Colt	Lawman Mark III		Speer	JSP	158
Colt	Python	6	RP		
Smith & Wesson	19	2-1/2	sv	Soft-nose lead SJHP	
Ruger	Black Hawk		Unknown	(.38 Spec.)	
Ruger	?		Unknown		
Unknown			Unknown		
. 380 Automatic Co	olt Pistol			<u> </u>	
Walther	PPK	3-1/2	R-P	FMJ	· · · · ·
Beretta	i e	3-1/2	Fed	FMJ	95
9mm	· · · · · · · · · · · · · · · · · · ·		<u></u>		
STAR		~ 3-1/2	R-P	FMJ	124
STAR			Fed	FMJ	
Luger		4	R-P	SJHP	115
Unknown		4	S& W	SJHP	115
.22 LR	<u></u>		•	· · · · · · · · · · · · · · · · · · ·	:
Rohm	66	5	Rem	RNL	40
Rohm	RG14	2	West	RNL	39
Rohm	RG14	2	Valor	RNL	39
Rohm	RG14	2	Winch.	RNL "Wildcat"	
Rohm	RG23	2	Rem. Mohawk	RNL	40
Rohm	RG ?	2	Rem	RNL "golden"	40
Ruger	Single 6		CCI Western	Lubaloy	
Ruger	?	5-1/2	R-P Cascade	RNL RN Lubaloy	40
High Standard	?	6	CCI	RNL	40
High Standard	?	2-5/16	Fed	HP, plated	39
F, I. E.	?	4	CCI(C)	RNL	39

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	Gun			Ammunition	
Make	Model	Barrel Length	Brand	Bullet	Weigh
		(in.)			(gr)
.22 LR					
General Precision	20	2	West Super X	RN, plated	-40
Reck	Single action	5	West Super X CCI (B)	RN, Lubaloy RNL	+0 +0
Colt	Peacemaker	7-1/2	RP Super X RP Standard	RN, plated RNL	
J.C. Higgins	88	2	Fed	RN Lubaloy	40
H&R	Nine shot		Browning		
H&R	?	2-1/2	Winchester, Super X	RN Lubaloy	40
S&W	61-2 (auto)		R-P	golden bullet	
Erma	(auto)		W-W long	Super X plated	
· ·			R-P long	plated	20
Unknown Unknown	(auto)	6	Fed Fed		- 39
Unknown	(rev)		Unknown		
. 22 Short		· · · · · · · · · · · · · · · · · · ·			1;
J.M. Marlin	?	3	Fed	plated	28
H&R	3		Western	Super X RN Lubaloy	
Rohm	Rosco	2-1/2	Rem	High vel golden	29
Rohm	RG-10	3	Western	RN Lubaloy	28
. 22 Magnum	••••••••••••••••••••••••••••••••••••••	······································	· · · · · · · · · · · · · · · · · · ·	·	
Rohm	66		Winch Magnum	SJHP (soft nose)	
Ruger	?	6	Winch Magnum	Super X SJHP	36
. 32 Smith & Wesso	n (Revolver Ammunition	n)	· •		
Ivers Johnson	Г	5	w-w	RNL	1
H&R	732	2-1/2	R-P	RNL	.88
H&R	?	2	R-P	RNL	

,

	Gun			Ammunition	
Make	Model	Barrel Length (in.)	Brand	Bulle Type	t Weight (gr)
32 Smith & Wesson	(Revolver Ammunition)	1 (<u>[</u>	
Rohm	RG ?	2	R-P	RNL	98
H& R	RG ?	2	Western	RNL	85
Liberty	H. W3 (7-shot)	2	w-w	FMJ	98
Clerk	1 st	snub- nose	R-P		
Unknown		-	Unknown		
Colt 32/20	a a	2		FN, plated	100
. 32 Automatic Colt	Pistol (Automatic Pistol	Ammunition)			
Unknown	Revolver, top-break	3	Fed	FMJ	70
Unknown	Auto-pistol			FMJ	70
,44 Magnum	<u>La popular en esta de la popular de la popular en esta de la popular en esta de la popular en esta de la popula</u>	-E			
Smith & Wesson	2	4	R-P	SJFN	1
Hawes	? ?	6	w-w	RNL (Rem-Mag)	
.45 ACP		•	- -
Colt	Gold Cup	5	Winchester Military	FMJ	230
.25 ACP				·	
Unknown		1	Unknown		Τ
Rohm	RG 25	~2-1/4	Rem	FMJ	50
Rohm	RG 25	~2-1/4	Fed W-W	FMJ FMJ	50 50
Rohm	RG 42	2-3/4	R-P	FMJ	50
Rigarm Brescia			Unknown	· · ·	
Raven Arms	P 25	~2-1/2	w-w	FMJ	50
F.I.E	Titan		R-P	FMJ	
Tanfaglio Guiseppe		2-1/2	R-P	FMJ	50
Shotguns	•	'			
Am. Gun Co.	.410 gauge	· ·		I	
Remington	12 gauge, model 870		W-W 00B	Shot shell	

Gun			Ammunition		
				Bullet	
Make	Model	Barrel Length (in.)	Brand	Туре	Weight (gr)
Shotguns					
Stevens	12 gauge	·····			[
?	12 gauge		Unknown (No	. 6 ?)	
Diamond Arms	12 gauge, Model 1905		Fed	Hi-power No.	7-1/2 shot
?	20 gauge		R-P		1
FI Industries	12 gauge		W-W	No. 6 shot	
Rifles	<u>6</u>	1 1			
Ruger	.22 cal, 10-22, semi-au	ito .	CCI	LR, plated	40
Remington	.22 cal, bolt action		CCI	LR	
Winchester	.22 cal, 67A, bolt action		Sears extra range	RNL	40
Glenfield	.22 cal, 65, semi-auto		Omark CCI	RN, plated	40
Glenfield	.22 cal, (short), servi-auto		Western	Super X, plated	40
Savage	.300 cal, 1899, lever a	ction	R-P	SP	~150
Ruger	.223 cal, Mini-14 carbi auto "LC70"	ne, semi-	Military spear point	(M-16), plated	
Marlin	30-30 cal, sawed-off, lever action Model 336 Rc			SJSP	55
Marlin	.22 cal, Model 81, lever action		Rem	.22 LR	Birdsh
Marlin	.22 cal ?		Fed	LR (probably)	

DEFINITIONS:

S&W:

Smith and Wesson, a manufacturer. The letters are also used to specify certain types of cartridges. For example, .38 S&W is a different type of cartridge than the .38 Special and is not interchangeable with it. The .32 S&W is a revolver cartridge, while .32 ACP is an auto-pistol cartridge.

ACP:

Automatic Colt Pistol. This designates cartridges for auto-pistols. The .380 ACP is equivalent to 9mm short; 9mm, sometimes called 9mm Luger or Parabellum, is a longer cartridge.

LR:

Long rifle. Twenty-two caliber rim-fire cartridges come in four increasing lengths, called: short, long, long rifle, and magnum.

H&R: Harrington and Richardson, a manufacturer.

R-P: Remington - Peters

W-W: Winchester - Western

SV: Supervel

Rem: Remington

CCI: Cascade Cartridge Inc.

WRA: Winchester Repeating Arms Co.

UMC: Union Metallic Cartridge

RN: Round Nose

RNL: Round-nose, lead

Lubaloy: A plating alloy for bullets SJHP:

Semi-jacketed hollow-point

LHF: Lead hollow-point

- +P:Indicates a high powder load, "extra pressure."
- WC: Wadcutter, a blunt bullet without taper

FMJ: Full metal jacket over a RN bullet

DEFINITIONS (Continued):

JSP:	Jacketed soft point
HP:	Hollow-point
SJFN:	Semi-jacketed, flat nose
FN:	Flat nose

material in the sample. Photographing particles or x-ray spectra takes additional time. The average for all samples in the cases that were handled was 1-1/4 hour per sample.

The number of samples per case also varies. The minimum is two, one from each hand of one suspect. There may be more than one suspect, more than two samples per suspect, and in most cases a sample from test firing the suspect's ammunition is submitted and analyzed also. Sometimes, not all samples that were submitted needed to be analyzed, but the average number actually analyzed in 69 cases was 4 samples per case, with a standard deviation of 2. One laboratory has increasingly used the method for screening large numbers of suspects. In three cases submitted by this particular laboratory, 8, 10 and 12 samples were analyzed, and this raises the average to 4.3 for 72 cases. Even larger numbers of samples were submitted in recent cases that have not yet been completed. Not all of the samples will be analyzed, however. The best procedure to follow for this purpose is to first examine a relatively small area of one sample from each suspect. If gunshot residue is found, it may not be necessary to spend much time on most of the rest of the suspects.

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CHAPTER VII. EQUIPMENT

A. Sampling Devices and Their Use.

The basic sampling device is a plain aluminum disk, one inch in diameter and about 3/8 inch high. It is cut from one inch bar stock, using alloys 6063 or 1100. These are the only aluminum alloys that are readily available in bar stock and are low enough in the heavier elements that are of interest in gunshot residue analysis.^a High-purity pyrolytic graphite can also be used.

The disks fit into a recess in the sample stages of some SEMs. Thin disks that have a stem or mounting pin on the underside are used with other SEMs. Two different pin diameters and a number of different stem lengths are commonly used. One-inch diameter pin-type sample holders can be purchased for about \$2 each. The plain 3/8 inch disks described in the foregoing paragraph can be made in any machine shop for between 20 and 50 cents each. For reasons of both cost and uniformity, it is advantageous to use the plain disks universally, and to use an adapter for those instruments designed for pin-type mounts. The adapter shown in Figure 9 is simple to fabricate, and only one is needed per instrument. The dimensions given in this illustration are for the AMR-1200 SEM.

It is not advisable to use diameters of less than one inch. The surface area is proportional to the square of the diameter, and sampling efficiency drops markedly if smaller diameters are used.

A one-inch square of Scotch Transfer Tape No. 465 is placed on the disk, adhesive side down. When the disk is to be used, the paper backing is pulled off, leaving the adhesive on the disk. Only the adhesive compound remains on the disk; there is no tape. Some adhesive tapes would do an

^aX-ray signals from elements in the disk are seen only through holes in the adhesive layer.


Figure 9. Adapter Cup for Sampling Disk

adequate job of sampling, but some blister and burn in the electron beam, and some contain impurities that can interfere with x-ray analysis. Many other tapes are not sticky enough, not smooth enough, or have inadequate shelf-life.

The limits of the shelf-life of Scotch Transfer Tape No. 465 have not been tested, but in the case analyses, disks have been used several months after preparation and have been mailed over considerable distances. In several cases, the disks were mailed to a destination 6000 miles away and were returned with gunshot residue on them and analyzed successfully. In some cases, analysis was delayed for four months after the samples were taken.

To sample the hand of an individual (see Figure 10), the disk is pressed



Figure 10. Use of the Sampling Disk

repeatedly against the hand, adhesive side toward the hand, and the disk is moved from place to place until it has lost its stickiness. The disk is pressed straight down and lifted -traight up. The disk is not to be slid or rotated on the skin, but it should be pressed down firmly.

The pattern of movement starts on the web area just behind the junction of thumb and forefinger. It is then moved along the backs of the thumb and the forefinger, the back of the hand further behind the web, the backs of the middle and fourth fingers, and finally the area far back of the hand. Sampling is stopped when stickiness is lost. An additional disk is used if more area needs to be sampled.

Sampling should be performed as soon as possible after the shooting, which is immediately upon apprehension of a suspect. The suspect should not be allowed to wash or rub his hands prior to the sampling. No fingerprints or swabs for other types of analysis should be taken prior to sampling.

If the suspect has washed his hands, they should not be sampled. Instead, his wrists and the sleeve above the wrist should be sampled. If several hours have passed between the shooting and the sampling, the hands, the wrists and sleeves, and the inside lips of the side pants pockets should be sampled, using separate disks. Normally, not all of these samples will be used; however, it is better to have them available in case they are needed.

Precautions should be taken to prevent contaminating the samples. Police officers conceivably may have residue on their hands from handling their own weapons. If they do the sampling, they should first wash their hands or wear gloves.

The adhesive-disk method of sample collection was developed originally in connection with the photoluminescence method of analyzing for bulk lead and antimony.⁵ It is easy to dissolve the residue on the disk in acid, provided the disk has not been coated with carbon. Therefore, the disk method is applicable to both bulk elemental analysis and particle analysis. In the

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work referred to in Note 5, a second sampling yielded less than 50 percent of the amounts of lead and antimony found in a first sampling, indicating a sampling efficiency of approximately 70 percent. In the sampling described in this report, sampling efficiency was further explored by counting particles collected in two successive samplings of the same hand. The two samplings gave 320 and 107 particles respectively, indicating that the first sampling collected 75 percent of the particles. Data published by Goleb and Midkiff¹⁸ indicate more than a 90 percent collection efficiency for regular Scotch adhesive tape.

In case work, sample disks are received in individual plastic boxes which are marked with the identification of the sample. The first step in the analysis is the transfer of the identifying legends to the underside of the disk with indelible ink. At this time, a mark is also made on the side of the disk. This allows it to be re-inserted into the microscope sample stage at a later date in approximately the same orientation as the one originally used, in case it is again necessary to find some of the same particles. The second step is coating the specimens.

For proper SEM performance, an electrically conducting path must exist from the sample surface to ground (the sample support). For this purpose, a coating of carbon is applied over the specimen. For materials that are subject to considerable outgassing, such as the adhesive, vacuum evaporation is preferable to sputter coating.^a A thin film of aluminum is acceptable in place of the carbon. For best picture quality, noble metals such as gold, rhodium, and palladium are often used. However, they are undesirable for x-ray analysis, because they diminish intensities due to their high x-ray and electron absorption, and they give rise to interfering spectra.

B. The Scanning Electron Microscope (SEM).

Several SEMs currently selling for \$30,000 to \$35,000 each are suitable for gunshot residue analysis, as are many higher-priced instruments.

^aTypical cost of a vacuum evaporator is \$5000. A sputter coater is cheaper.

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This price includes all of features mentioned in this chapter except the x-ray system. The nominal resolution should be of the order of 100 Angstrom Units (10 nanometers). This requirement currently excludes only those instruments that sell for substantially less than \$30,000.

Except for the vacuum system, the principal mechanical component of a SEM is the sample stage, which allows the specimen to be tilted, rotated, and moved along x, y, and z axis directions. The workmanship required for this stage exceeds that found in high-quality mechanical watches. When viewed at magnifications of several hundred to several thousand times, any unevenness of the x and y motions will manifest itself as an erratic jumping of the specimen across the field of view, making it difficult to center an object of interest and impossible to conduct a rapid systematic search over a large area. When contemplating a purchase, the manufacturer's ability to meet this requirement should be ascertained. Any stage that is deficient should be rejected.

The instrument must be capable of providing an accelerating potential of up to 25 KV, either continuously or in steps. Most specimens require no more than 10 to 15 KV and sometimes less for good picture quality. Specimens prone to damage by the electron beam should be viewed at the lowest feasible voltage. However, a potential of 25 KV is essential to performing an adequate x-ray analysis. The definition of "adequate" is the excitation of x-ray emissions in the energy range from 1 to 20 KEV with sufficient intensity. Operating at 25 KV instead of at lower voltages does not degrade picture quality. It provides maximum resolution, but it leads to faster contamination of the column and apertures, especially in the presence of organic material, such as the adhesive, necessitating more frequent cleaning.

The instrument must be equipped with a TV scan capability. In the majority of lower-priced scopes, this is an optional extra-cost feature, but

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it is included in the \$30,000 to \$35,000 quoted. If the manufacturer states his fast scan to be TV tape recorder compatible, he has a true TV scan. In some instruments, a simulated TV scan performs as well, but quite often it is not satisfactory.

For normal viewing, images are generated at scan rates typically in the neighborhood of one frame per second. Even slower speeds are used for photography. To create satisfactory images at such slow scan rates, the phosphors which are activated on the face of the cathode ray tube used for viewing must have an appreciable persistence. As a consequence, when the specimen is moved, one image will slowly fade at the same time that the new image is gradually built up. When rapidly searching over an extended area, the specimen is moved continuously. This requires a much faster scan rate and a phosphor with shorter persistence. A true TV scan produces 30 frames per second and is interlaced. In this mode, the picture is usually displayed on an extra (external) monitor scope, which quite often is actually a small black and white TV set. (It is possible to use a large set, or several connected in parallel). The picture quality is degraded because the number of lines per inch is reduced, but a rapid search is possible. A few SEMs will display the TV scan on the same scope that is used for normal viewing. The adequacy of such a feature should be checked very carefully.

Another essential feature is the ability to operate the TV scan at all magnifications. On some instruments, the TV scan is operable only at the lower magnifications, and the manufacturers will point out that the TV image becomes too washed out at high magnifications to be useful. However, particle analysis employs a special operational procedure which has its own requirements.

In the particle analysis operational procedure, the specimen is searched in a systematic pattern, using the TV scan only, at a nominal magnification of at least 500X, but not much more. On the instrument used in this program, the TV image automatically had a magnification over twice as large. However, this is secondary (empty) magnification, which is comparable to using a more powerful ocular, rather than objective, in an optical microscope. Under these conditions, the smallest object just visible is about 1/2 micrometer in diameter. When the operator spots a particle that may be of possible interest because of its shape and brightness, he increases the magnification to get a better look. If the particle still looks interesting, he turns the magnification up until the particle fills the screen (keeping it centered) and presses the button that initiates an x-ray analysis which will identify the particle either as gunshot residue, possibly gunshot residue, or not gunshot residue (or whatever else for which one might be searching). At this high magnification, the TV picture loses all visual detail, but the purpose is to keep it centered for the x-ray analysis without switching out of the TV mode.

If the TV scan cannot be operated at high magnifications, it is necessary to return to the normal viewing scope, and either a slow scan or perhaps a spot mode before taking the x-ray spectrum. This procedure must then be reversed to continue the search. All this is time-consuming; if there are many particles, the analysis may take five times as long as it would in a continuous TV mode operation. The ability to operate the TV scan at all magnifications is essential for particle analysis.

In general, when switching from normal to TV scan rates, the diameter of the electron beam (spot size) needs to be reset in such a way that the energy input to the sample is increased. Different instruments differ in the amount of change that is necessary, and the less change required the better are the results. The increased energy is more damaging to specimens and, in the case of adhesive lifts, is more prone to melt or decompose the adhesive and allow the particles to sink out of view.

C. The X-Ray Analysis System.

For small and irregular-shaped particles, it is usually a waste of time to engage in elaborate treatments of SEM x-ray data in an attempt to

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obtain quantitative analyses. Automatic rather than operator identification of the elements that are present is an expensive convenience that does not speed the analysis if the operator is experienced; it may in fact slow the analysis. For this reason, for gunshot residue examinations, a relatively simple x-ray analysis system is preferred with just a few of the many options available. Specifically, a middle-of-the-line system in close to its basic configuration is recommended. Currently, this can be bought for approximately \$22,000.

The detector resolution must not be worse than 152 eV, which is the current industry standard (lower numbers mean better resolution); 145 eV detectors are available at considerably higher costs. When these prices are reduced, they should be used. The analyzer should have a memory of at least 1024 channels and the ability to put element markers (MLK markers) into the display. The display should have an energy scale along the bottom, and it should be possible to expand and contract the scale, as well as move it to the right and left. The display should be of adequate size, at least a nominal 9-inch (diagonal) TV or cathode ray tube (CRT), and conveniently positioned. Systems should be avoided in which the display consists of a small CRT-mounted integral with a large electronic bin. These are both hard to read and difficult to position for operational convenience. Scanning electron microscopy is a steady, hour-after-hour occupation, in which operator convenience is important.

One of the components of an x-ray analyzer is an electronic module usually called a pulse processor. It is useful to have a pulse pile-up rejection feature incorporated in an x-ray analyzer. Further options and capabilities are not required for gunshot residue determinations, but should be considered on merit for other possible applications before a purchase is made. Occasionally, some of these features can be useful for gunshot residue as well (see Chapter IX).

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CHAPTER VIII. STATISTICAL ANALYSIS OF RAPID SEARCH AND CHARACTERIZATION PROCEDURES

A. The Sample and the Instrument.

1. <u>Geometry</u>. The large circle shown in Figure 11 represents the surface of the cylindrical sampling disk, which is one-in. (2.54 cm) in diameter. The surface area is 5.07 cm^2 , or 0.7854 in^2 . The 0.75-in. square (20mm on edge) represents the area accessible for viewing by using





the x and y axis stage motion controls. Both its size znd location with respect to the center of the disk will differ for different instruments, and are shown in this illustration only for the AMR-1200 SEM. The location with respect to the center is affected by the tilt of the specimen from the horizontal toward the detector. Rotating the sample disk under this square allows the entire surface area of the sample to be observed. However, since the fraction of the circle lying within the square in a single orientation is larger than the area normally surveyed for gunshot residue, the disk is not rotated.

All searches are confined to a portion of the shaded area only. The shaded area is 0.40 in. x 0.75 in. (about 10 \times 20mm) in size and represents 38 percent of the disk area.

The shaded area is divided into strips. These are columns (y-coordinate) in the instrument used in this project, but may be rows (x-coordinate) in others. The width of a column, 0.008 in. (0.20mm), is the width of the field of view of the TV scan monitor at the nominal magnification of 500 used for the rapid search. On the instrument used in this project, this nominal magnification refers to the photographic scope. The visual scope and the TV monitor give pictures that are actually 1.3 and 2.6 times larger, respectively, than the photographic image, so that the search is actually performed at a magnification of 1300, but the resolution of a 500x picture.

Each column is scanned by first exercising the y-coordinate control, then a new column is selected by using the x-coordinate control until enough area has been searched to meet the objectives of the experiment.

Each column covers about $4mm^2$, or 0.763 percent of the disk area, which corresponds to the equivalent of about 131 columns. There are 50 columns (about 40 percent of the disk within the shaded area.

The disk area surveyed is always less than 40 percent. However, no matter how large or how small the area actually surveyed is, it is never one contiguous area. It is made up of several portions scattered randomly over the (accessible) area of the rectangle.

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The y-coordinate is measured in a horizontal plane. Since the disk is tilted downward by 10 degrees toward the detector, the actual y distance along the surface of the disk is 2 percent larger than the measured distance (y/cos 10°). This is not significant within the rounded-off figures used.

B. Probability of Detection in Rapid Search Procedures.

The following two questions are answered in the paragraphs which follow: (1) If no gunshot residue is found in the initially surveyed portion of a sample, how much more area needs to be examined before there is a satisfactorily high probability that nothing would be found in the remainder? (This question is important because the time required to scan 100 percent of the area would be prohibitive in most cases.) and, (2) In characterization studies, what percentage of the area is sufficient to allow statistically valid extrapolations for the totals?

"Clustering" does not occur.^a The method of collecting residue and moving the adhesive-coated disk from place to place causes some randomization of the deposits. More importantly, in literally hundreds of examinations, no situation has been observed in which frame after frame was empty when viewed on the TV scan monitor, when suddenly one was found having five to ten particles in the field of view. If the specimen is sparse, an occasional particle is seen in isolation here and there; there are no closelyspaced groups of separate particles separated by large empty stretches. If the specimen is rich, then it is rich everywhere, although the number of particles per column will vary. Some data on this variation are presented in VIII. C. The distribution of particles on the disk is random, and there is no tendency of particles to cluster.

^aThe term "clustering" is used here in the sense of a close grouping of independent particles. It does not refer to the clusters described in II. B, which consist of connected spheres and are counted as single particle of a special nature. The total area of the disk is equivalent to 131 columns (see VIII. A.) Assuming that the deposit is rather sparse and consists of only 25 particles on the entire disk, with random distribution of the particles and given the absence of clustering, the simplifying assumption can be made that there are 25 columns which contain one particle each and 131 - 25 = 106 columns that are empty. Thus, the probability of finding any column empty is 106/131, or 0.809. If the first column searched was found empty, the probability of finding another column also empty is 105/130, or 0.808. The combined probability of finding both empty is the product of the separate probabilities, or (0.8)² = 0.66.

Continuing in this fashion, the following probabilities are found, with the superscript giving the number of columns searched:

 $Pe^{1} = 0.81$ $Pe^{2} = 0.66$ $Pe^{10} = 0.11$ $Pe^{20} = 0.001$ $Pe^{30} = 0.00001$

The meaning of these figures is that there is only a 0.1 percent probability of finding the first 20 columns empty if there are 25 randomly distributed particles on the disk. If 20 empty columns are found, either an event of low probability has been produced, or else the original assumption that there are 25 particles is wrong. The interpretation can therefore be turned around and the inference made that if no particles are found in the first 20 columns examined, there is only a 0.1 percent probability that there are as many as 25 particles in the remainder of the area. Similar calculations can be made for different assumed numbers of particles. The results are listed in Table 10, and the program that produced these numbers is listed in Appendix B.

	· · · ·		. <u> </u>		
Particles->	5	10	15	20	25
Columns		·	Probabilit	ÿ	
1	0.96	0.92	0,89	0.85	0.81
10	0.67	0.44	0.28	0.18	0.11
20	0.30	0.085	0.02	0.006	0.001
30	0,083	0.006	0.0004	2.3×10^{-5}	1×10^{-6}
40	0.013	0.0001	$1 \ge 10^{-6}$	9×10^{-9}	5×10^{-11}
50	0,001	1×10^{-6}	6×10^{-10}	3×10^{-13}	6×10^{-17}

Table 10. Probability that the Unsearched Portion of the Disk Contains the Indicated Number of Particles as a Function of the Number of Columns Searched

In this project, an upper limit of 25 percent was set on the area to be surveyed. Twenty-three percent would be equivalent to 30 columns. Using the figures for 30 columns, if nothing has been found, there is an 8 percent probability that there may be five particles on the remainder of the disk, a 0.6 percent probability that there may be 10 and a 0.04 percent probability that there may be 15. Using the 50-column maximum area accessible without rotating the disk still leaves a 0.1 percent probability of finding five particles, but a negligible probability of finding more than five.

In the event that clustering occurs, assume an extreme case in which 25 particles are distributed in such a fashion that they are contained within only five columns, each of which would then contain five particles. Obviously, the correct probabilities are now those given for 5 rather than 25 particles in Table 10. Such extreme clustering is known not to occur, but a random rather than a uniform distribution does allow for a very

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limited amount of clustering. For any number of particles, a safe procedure is to use the figures in the column for the next lower number of particles. For example, use the 15 particle column for 20 particles.

The cartridge characterization studies reported in Chapter III and the persistence studies reported in Chapter IV, give a general expectation of the number of particles to be found if the subject fired a gun. Although these expectations are probabilities rather than certainties, they furnish a standard by which the progress of an analysis can be judged. If the analysis has progressed to the point where there is only a small probability of finding at most a small fraction of what was expected, it should be determined whether spending any more time on the examination is justifiable.

C. Reliability of Extrapolations in Characterizations.

This section is addressed to determining the percentage of the disk area that must be surveyed to allow statistically valid extrapolations to determine the total number of particles at a predetermined level of confidence. This will depend on the fraction of the total area over which counting occurs, and the scatter of particle counts from column to column.

A relationship for this confidence level can be derived from statistical considerations. It applies with increasing reliability as the total number of particles increases. Figure 13 illustrates the approach taken and the meaning of the symbols used.

The total number of columns on the disk is Z, and individual particle densities (n) have been determined for z columns (which need not be adjacent). The mean particle density for the z columns is $\overline{n_1}$. Z-z is the number of columns that remain uncounted and in which the mean particle density will be $\overline{n_2}$. The total number of particles is then:

$$N = \overline{n}_1 z + \overline{n}_2 (Z - z)$$
(1)

Because of the collection method, it is probable that the particles have a random distribution over the disk. This has been confirmed experimentally. In this case, the most probable value for \bar{n}_2 is \bar{n}_1 ; i.e., the possible values of \bar{n}_2 are represented by a distribution curve centered about \bar{n}_1 . The width of the distribution curve can be expected to be determined by the degree of scattering of the individual values of n about \bar{n}_1 . If a standard deviation:

$$\sigma = \left(\frac{\sum_{i=1}^{z} (n_{i} - \bar{n}_{1})^{2}}{z - 1}\right)^{1/2}$$
(2)

has been obtained for the counted area, it can be inserted into a Gaussian distribution for \overline{n}_2 according to:

$$\overline{n}_2 = \overline{n}_1 e - \frac{(n - \overline{n}_1)^2}{2\sigma^2}$$
 (3)





The probability of finding \overline{n}_2 within the limits $\pm k\sigma$ is obtained by integrating Equation 3 and normalizing the total area under the curve so that:

 $\int_{-\infty}^{\infty} = 1$

This yields:

$$P(k\sigma) = \frac{1}{\sigma\sqrt{2\pi}} \int_{-k\sigma}^{+k\sigma} e^{-\frac{(n-\overline{n}_1)^2}{2\sigma^2}} dn \qquad (4)$$

The probability of finding the overall particle density N/Z (and hence N, the total number of particles) within the same limits after counting only z columns, then follows from combining Equations 1, 3, and 4:

$$P(z/Z, k\sigma) = \left[z/Z + \frac{Z-z}{Z} P(k\sigma)\right]$$
(5)

Equation 5 has been evaluated for its dependence on z/Z and $k\sigma$, and the results are shown in Figure 13. It is seen that P increases in proportion to the number of columns counted. For z = Z, P = 1 for all values of k. If the range of variation for N is taken as wide as $\pm 3\sigma$, N can be found within this range with better than 99 percent probability, even if only a rather small fraction of the total area has been surveyed. It should be kept in mind, however, that σ is poorly defined if $z \ll Z$, and the task remains of deciding how large z/Z needs to be so that σ is adequately determined.



Figure 13. Plot of Equation No. 5

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Figure 14 is a plot of nine tests in which from 10 to 25 (average 18) columns were counted for their particle content. The fractional variation from column to column, in the form $\sigma_f = \sigma/\bar{n}$ where \bar{n} is the mean number of particles per column, is plotted against \bar{n} . Not unexpectedly, σ_f declines with increasing \bar{n} . The fact that it does so with only a modest degree of scatter suggests that σ is meaningful for 18 columns if \bar{n} exceeds 3.



Figure 14. Fractional Standard Deviation as a Function of Mean Particle Density

Table 11 shows a determination in quadruplicate (four separate firings by different persons) of the residue left on the firing hand by a particular cartridge. Particles were counted for each of the four samples, first in 5 percent, then in 10 percent, and finally in 15 percent portions of the total area and the numbers were extrapolated to 100 percent. With the addition of more area, the changes in the extrapolated numbers are consistently smaller than the sample-to-sample variations. Therefore, it is the latter rather than the former which determine the standard deviation of the result, and the numbers derived from only 5 percent of the area would have been acceptable. If that is true for this fairly sparse residue, then because of the data shown in Figure 14 it should be even more valid for heavier deposits. It also confirms the previous conclusion that the residue is distributed fairly uniformly over the disk.

Percentage		Mean and σ			
of Area	1	2	3	4	
		Particle Count			
5%	180	200	180	80	160 <u>+</u> 54
10%	140	210	130	80	140 <u>+</u> 54
15%	140	193	133	107	143 <u>+</u> 36
Mean and $\boldsymbol{\sigma}$	150 <u>+</u> 23	201 <u>+</u> 9	148 <u>+</u> 28	89 <u>+</u> 16	

Table 11. Extrapolations from Increasing Fractions of Area

The data in Table 11 can be evaluated further to extract numbers for use with Figure 13. Instead of examining the change in the extrapolated totals as the area increases from 5 to 10 to 15 percent, a column by column count is made for 15 percent of the area and $\overline{n_1}$, the average number

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of particles per column, and a standard deviation are obtained. The latter is the standard deviation for each column. To obtain the standard deviation for the entire area examined, if two numbers are added, each of which has a certain standard deviation, the σ for the sum is the square root of the sum of the squares of the individual sigmas, thus:

$$\sigma_{A+B+C} = \sqrt{\sigma_A^2 + \sigma_B^2 + \sigma_C^2}$$

The results, multiplied by three, are listed in the row labeled 3 x cumulative σ in Table 12. In accordance with the data in Figure 13, there is a 99 percent probability that the extrapolated number is correct within this tolerance.

Sample	1	2	3	4
No. of Columns	20	19	17	20
Percentage of Area	15	15	13	15
n	1.05	1.52	0.95	0.90
σ	0.95	1.21	1.38	0.86
$3 \ge Cumulative \sigma$	13	16	17	12

Table 12. Derivation of Standard Deviations

To check the degree of uniformity of the deposits further, the effect of increased area coverage on the extrapolated totals was examined for five cases in which the totals ranged from 3000 to 10,000 particles. In three cases, increasing the area from 1.5 percent to 3.0 percent resulted in changes by factors of 0.91, 0.96, and 0.91. In one case, going from 2.3 percent to 4.6 percent gave a factor of 0.77, and the last of these five cases gave a factor of 0.92 for a change from 2.3 percent to 10.0 percent. A sixth case with a total of 2000 particles changed by 0.56 for an increase in area from 1.5 percent to 15.0 percent. Even this comparatively large change is well within the sample-to-sample variations.

It is concluded that examination of 5 percent of the total area is a safe procedure for estimating the total number of particles, within a factor of two, and that 10 percent can be expected to provide reliable statistics on the distribution of the particles among the various size and composition classes, provided that the total number of particles is large enough for statistical purposes. These percentages were exceeded for most of the characterizations described in this report, 15 percent being the most commonly used figure. Accordingly, the standard deviations found are primarily those of the sample-to-sample variations.



CHAPTER IX. X-RAY ANALYSIS

A. Quantitative Aspects.

X-ray emission in the SEM is induced by electron excitation. (See Table 13 for a comparison of electron excitation with x-ray excitation methods.) The detection method that is the most commonly used for SEMs employs a solid state detector and energy-dispersive analysis of the spectra. In this method, the relative peak heights of the characteristic emission lines are not directly proportional to concentrations. For flat specimens, the peak heights can be converted to concentrations by fairly complex mathematical procedures that are usually performed by computers. A simple comparison of peak heights will give reasonably quantitative results only if the specimens are large and flat, and if similar specimens of known concentrations (standards) are available. For small and irregular-shaped specimens, reliable quantitative analyses cannot be obtained in the SEM by any method.

Detection Method Salient	Excitation by:						
Characteristics	Electrons	X-rays					
Energy-Dispersive Detection: Low resolution, High sensitivity. Quantitative analysis is difficult	SEM	X-ray Microprobe					
Wavelength Dispersive Detection: Higher resolution (100X), Lower sensitivity (1/100 to 1/10). Easier to make a quantitative analysis.	Electron Microprobe (SEM if specially equipped)	X-ray Fluorescence					

Table 13. Comparison of Analysis Methods

Only a simple qualitative detection system was used in this project. The classification of elements as major, minor, and trace constituents is based on peak heights rather than on concentrations. Since the peak heights are modified by irregularities of shape and by matrix effects, even these broad distinctions cannot always be taken literally. With this reservation, elements whose strongest peak has 30 percent or more of the height of the strongest peak in the spectrum are deemed to be major ingredients. Minor constituents are elements whose strongest peak is smaller than this but is clearly identifiable when the strongest peak in the spectrum is still on scale. The term trace is used when the element can only be seen or identified by enlarging the vertical scale or continuing the x-ray count until the strongest peak is well off scale at the top of the display. In general, this means that the peak height is of the order of 1 percent of the strongest peak height, and the element is just detectable above the background. The term just detectable is a better description than trace. The detection limits of this method range from a few hundredths of one percent at the very best to several percent at worst, whereas in the general field of analytical chemistry trace analysis refers to the determination of concentrations smaller than 500 parts per million down to parts per billion. However, the modest concentration detection limit of 1 percent translates into absolute amounts of this component on the order of femtograms $(10^{-15}g)$ when a particle one micrometer in size is analyzed. Trace is defined as meaning just detectable by the methods described in this chapter.

B. Energy-Dispersive X-Ray Analysis Problems.

1. Extra lines, artifacts. The absorption by the silicon detector of any strong x-ray with an energy greater than that of the silicon K α line may cause the emission of a silicon K α x-ray which will appear in the spectrum, together with an escape peak whose energy will be the difference between the energy of the exciting line and that of the silicon K α (1.74 keV). The smaller this difference is, the greater is the likelihood for observing this phenomenon. If strong, the unresolved lead M α lines (2.34 and 2.35 keV) are accompanied regularly by the 1.74 silicon K α line and an escape peak at 0.606 keV. The operator should become acquainted with the intensity ratio between the lead $M\alpha$ and the spurious silicon $K\alpha$. An increase in the intensity of the latter indicates the actual presence of silicon in the sample. The operator should also be aware of the possibility that other strong peaks can cause silicon escape phenomena. A peak that appears in the spectrum below about 1.0 keV is a priori an escape peak, because if x-rays of such low energy originated outside the detector (in the sample), they would not penetrate a conventional detector window with sufficient intensity to be registered. For example, the cadmium $M\alpha$ line has the energy of the lead-silicon escape peak, but would not be seen in the spectrum.

At high counting rates, a phenomenon known as Pulse Pile-Up can cause low intensity peaks at energies that are either twice that of a strong peak or the sum of two strong peaks. The better detector systems have circuitry to prevent this from happening.

In the work described in this report, the only spurious line observed was the silicon $K\alpha$ and its escape peak in the presence of a high concentration of lead.

2. <u>Extra lines, real.</u> Analysis of spectra is made both easier and faster by the ability of the detection system to place element markers (MLK markers) on the screen at the positions where the major lines of an element occur. Generally, this is limited to two K, four L and one M line for one element at a time. Tables supplied by the manufacturer or found in books list additional lines frequently observed. However, x-ray spectra are richer than is commonly believed. There are many more weak lines not listed in the usual references, and some of these may be occasionally observed. Since all lines in a spectrum must be accounted for, the operator should equip himself with an exhaustive listing such as ASTM publication DS-46, by Johnson and White, entitled X-Ray Emission Wavelengths and keV Tables for Non-Diffractive Analysis.

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In this project, the following lines, which are not listed in abbreviated tables, were observed:

- The barium Ll line at 3.96 keV, with an intensity of 2 percent of L α . This falls between the antimony L β_3 at 3.93 and the antimony L β_2 at 4.10. The antimony L β_6 at 3.98 is probably too weak to be important.
- The lead $M_2 N_4$ line at 3.124 keV, ^(a) with an intensity of 5 percent of $M\alpha$ claimed, it appears weaker. This overlaps the cadmium $L\alpha_2$ at 3.126 (unresolved from the cadmium $L\alpha_1$ at 3.133). Ensure that the remaining cadmium L lines are absent before deciding that the lead line is visible.

The extra barium line complicates the resolution of the calcium K and L lines that crowd one another in this region (see IX. B. 3). The extra lead line is observed when the lead $M\alpha$ is allowed to rise well above the top of the scale in order to detect a trace of antimony.

Other additional lines are the barium L_{γ_1} and L_{γ_2} lines at 5.53 and 5.80 keV, with intensities of 10 percent and 2 percent of the $L\alpha$, and a lead Ll line at 9.19 keV with an intensity of 2 percent of the $L\alpha$.

Quoted intensity ratios can vary considerably for irregular particles, or particles that are not homogeneous.

3. Resolution of overlapping peaks.

a. <u>Calcium and antimony</u>. Calcium is recognized by its K α and K β lines at 3.69 keV and 4.01 keV, respectively, as shown in Figure 15. Antimony is identified by its L α line at 3.60 keV, L β_1 at 3.84, and L β_2 at 4.10, as shown in Figure 16, which also displays the weak L ℓ at 3.19 keV. Because of the overlap of the calcium K spectrum with the antimony L spectrum, the analysis becomes difficult when both elements are present. Figures 17 through 20 illustrate the line shapes and peak positions for various

^(a)This line has not been assigned a spectroscopic symbol and is therefore designated as the line due to the transition between the M_2 and N_4 energy levels, or the $Pb_{M_2-N_4}$ line for short.



Figure 15. Calcium X-Ray Spectrum



Figure 16. Antimony X-Ray Spectrum

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Figure 17. Antimony : Calcium X-Ray Spectrum = 2:1



Figure 18. Antimony : Calcium X-Ray Spectrum = 1:1



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Figure 19. Antimony: Calcium X-Ray Spectrum = 1:2





Figure 20. Antimony: Calcium X-Ray Spectrum = 1:8

mixtures of calcium and antimony. These spectra were produced in the following manner. Samples of pure antimony, calcium fluoride, and barium oxide were placed on separate locations on the same sample support disk. Values of magnification were determined (this selects the size of the area from which x-rays are obtained) for each material, at which the heights of the strongest peaks for the three materials were equal to one another for equal counting times. To obtain the spectrum shown in Figure 17 in which the contribution of antimony is stated to be twice that of calcium, an x-ray count from the antimony was accumulated for 16 seconds. The count was then stopped and the calcium sample moved under the electron beam. The magnification was adjusted to the predetermined value and an additional 8-second x-ray count was accumulated, leading to the combined spectrum seen in Figure 17. In this manner, it is possible to specify the contributions of each material to the total signal, but the ratio of signal contributions is not the same as the concentration ratio corresponding to it, which was not determined.

In Figure 17, the addition of calcium has modified the antimony spectrum, but the three characteristic antimony L lines remain identifiable. Figure 18 shows the spectrum of a calcium-antimony mixture with equal signal contributions. The L β lines for antimony in this illustration are barely visible as shoulders on the main peak, whose energy maximum has been shifted to a slightly higher energy than the correct value of 3.60 keV for the antimony L α . In Figures 19 and 20, the calcium contributions are, respectively, twice and eight times those of antimony. The major peaks in both these spectra have maxima close to 3.7 keV, the energy of the calcium K α line. However, the good resolution of the K α and K β lines that are obtained with pure calcium does not exist, because the valley between the peaks has been partially filled in by the weak antimony lines.

b. <u>Calcium and antimony in the presence of barium</u>. Barium is recognized mainly by its $L\alpha$ at 4.46 keV, $L\beta_1$ at 4.83, $L\beta_2$ at 5.16, and L_{γ_1} at 5.53 keV. When the barium signals are strong, the weak $L\ell$ line at 3.95 keV is seen also, as shown in Figure 21. The presence of the $L\ell$ line complicates the detection of smaller amounts of calcium and antimony, which is a very common situation. The spectra in Figures 22 through 24 illustrate

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Figure 21. Barium X-Ray Spectrum



Figure 22. Barium : Antimony X-Ray Spectrum = 30:1



Figure 23. Barium : Calcium X-Ray Spectrum = 30:1



Figure 24. Barium : Antimony : Calcium X-Ray Spectrum = 60:2:1



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Figure 25. Barium : Antimony : Calcium X-Ray Spectrum = 60:2:2



Figure 26. Barium : Antimony : Calcium X-Ray Spectrum = 60:1:2

various mixtures of calcium and antimony in the presence of an excess of barium. Figure 22 represents a small amount of antimony but no calcium. Note the antimony $L\alpha$ at 3.60 keV. Figure 23 shows the reverse, i.e., no antimony; the calcium $K\alpha$ is seen at 3.7 keV. In Figure 24, both minor ingredients are present, and the antimony contribution is twice that of calcium. The peak close to 3.6 keV is indicative of antimony. This peak shifts toward 3.7, and the valley at about 3.8 also shifts to higher energies as the calcium ratio in the mixture increases. In Figure 25, the calcium and antimony contributions are equal, and in Figure 26 the calcium signal is twice as strong as that from antimony.

c. <u>Titanium and barium</u>. With a detector resolution of 170 keV or worse, titanium and barium may constitute a problem in resolution analogous to that posed by calcium and antimony. However, the peaks are resolvable with a detector resolution of 152 keV, which is the current industry standard.

d. <u>Lead and sulfur</u>. The lead M spectrum contains a number of lines, but the only strong ones are unresolved from one another and form a single, broad peak at. 2.4 keV. The L spectrum has its principal peaks at 10.5, 12.6, and 14.8 keV.

The only peak available for the identification of sulfur is the unresolved K line at 2.4 keV. In the presence of lead, it is covered by the lead M peak. To decide whether a peak at 2.4 keV is due to sulfur or to lead, the lead L lines should be sought. If these are absent, the 2.4 keV peak is sulfur. If they are present, the 2.4 peak is either lead or a mixture of lead and sulfur, and the presence or absence of sulfur cannot be decided.

e. Instrumental solutions to the problem of overlapping lines. Some of the optional, extra-cost features of x-ray analysis systems could be useful when problems arise like those discussed in IX. B. 3. c and d. One possibility is a computer-based deconvolution of spectral line profiles to determine if such profiles are made up of contributions from more than one

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line. Another possibility of solving the problem is through the use of the following options:

- Storage of experimental spectra in additional memory or on tape for later recall,
- Generation of theoretical spectra (which can also be stored), and
- The ability to subtract stored spectra from new experimental spectra.

These capabilities could be used if it is suspected that in an experimental spectrum the lines of element A overlap and obscure lines from element B. A stored spectrum of pure A would be subtracted from the experimental spectrum, and if B were present, it should then be visible. However, it can be envisioned that some careful adjustment of the relative intensities of the two spectra may be required to make this method work properly. In order to assess the practicality of this procedure, some experiments were performed with an x-ray system that was equipped with these features. The intensity of stored spectra can be multiplied before subtraction by arbitrary factors, both larger or smaller than unity.

The conclusions reached on the basis of these experiments are that subtraction of spectra gives unambiguous results in a controversy involving calcium and antimony, but a much more tentative conclusion when lead and sulfur are involved.

To understand this difference in results, it must be recalled that the calcium K and antimony L spectra have different numbers of lines. Where these overlap, they do not do so exactly but only because of their widths. Under these circumstances, subtracting increasing intensity contributions of one component from the combination allows the spectrum of the other component to emerge and be recognized because it is different.

Figure 27 shows the x-ray spectrum of a mixture of calcium and antimony in a 1:1 intensity ratio, similar to Figure 18. The end result of subtracting more and more of the calcium contribution is the antimony spectrum shown in Figure 28.

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Figure 27. Antimony : Calcium X-Ray Spectrum = 1:1



Figure 28. Antimony-Calcium Mixture (at the left) After Subtrac-tion of Calcium Antimony Line Markers Shown

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Figure 30. Sulfur X-Ray Spectrum










Unlike the situation in the case just discussed, the lead M and sulfur K peaks are indistinguishable within the resolution of the instrument. If increasing intensities of lead are subtracted from a mixture of lead and sulfur, there is no way to recognize the point at which all of the lead contribution has been cancelled and the remainder is due to sulfur. It may be thought that this could be accomplished through the use of the lead L lines. If lead intensity is subtracted until the L spectrum is reduced to background levels, as shown in Figures 29 through 32, is the lead M line likewise down to background? The answer is that this may be true but one cannot be certain of it. For all elements, the intensity ratios between the K, L, and M spectra are subject to alteration by a complex inter-play of many variables which include but are not limited to sample geometry and matrix effects.

These problems of line overlap do not exist with wavelength dispersive analysis systems because their resolution is better by two orders of magnitude. They are less sensitive and, because they take up more space in the sample chamber, cannot be installed in some instruments. They are also more costly. Some of the most advanced (and costly) SEMs are equipped with both energy dispersive and wavelength dispersive systems, one for sensitivity and the other for resolution.



CHAPTER X. ALTERNATIVE PROCEDURES

A. Concentration Separation Experiments.

Before a considerable body of case experience existed, there was concern that very sparse deposits of gunshot residue might require excessively long examination times because of: (a) the large fraction of the sample area that might have to be searched, (b) the time consumed in looking at and rejecting large numbers of extraneous particles to find the few gunshot residue particles dispersed among them, and (c) in the early phases of the project, the use of a SEM that was less suitable for this procedure than the one presently used and which consequently took much more time.

These concerns are now considerably diminished because of: (a) the high success rate experienced in cases (at least in those involving handguns), (b) the statistical calculations of the probabilities for finding particles described in VIII. B and VIII. C., and (c) the use of a more efficient SEM.

In the light of the earlier considerations, a project was initiated to develop methods to remove the residue from the surface of the sampling disk and re-deposit it on a smaller area in the center of the disk, and in the process remove as much of the extraneous material as possible. While subsequent experience has indicated a lessor need for such a method than the initial estimate, it would still be useful if it also were relatively simple and fast. The gunshot residue detection success rate for long guns is not as high as it is for handguns and it might be improved by concentrating the residue. Sampling of residue on fluffy or deep-pile fabrics puts so many fibers into the sample that gunshot residue particles become difficult to see. Separating the fibers from the residue would be beneficial.

In addition to fabric fibers, much of the extraneous material in the samples consists of skin debris and hair. All these are of sufficiently low density to remain suspended in organic solvents with densities near 1.5 g/l, such as trichloroethylene. On the other hand, gunshot residue particles are much denser since they consist of lead and inorganic compounds of heavy elements.

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A heavily cross-linked polymer which is insoluble in any reasonable choice of solvent is used in Scotch Transfer Tape 465. In order to carry out the following experiments, it was replaced on the sampling disk with an organic-soluble, non cross-linked poly-butylacrylate adhesive.

After sampling, the adhesive with the residue that had adhered to it was dissolved in trichloroethylene and the resulting solution centrifuged. The supernatant liquid, with extraneous material dissolved or suspended in it, was decanted, and the precipitate was transferred by Pasteur pipette to an area less than 4 mm in diameter on another sampling disk for SEM analysis.

Tests were carried out in parallel for direct comparison of this method with the normal manner of analysis. It was found that the use of this separation/concentration procedure reduced the time required for analysis by as much as a factor of five. Unfortunately, only 5 percent of the originally present gunshot residue was recovered by this procedure. Substituting filtration through Micropore filters with a $0.45 \,\mu m$ pore size for the centrifugation step resulted in comparably poor recoveries. Presumably, many of the gunshot residue particles were preferentially adsorbed on the glassware used in the various steps. Although effective in removing background material, in its present state of development this method is not suitable for routine forensic application because of its inefficiency in recovering particles and the length of time required to carry it out. While the problems encountered may be soluble through further efforts, it is clear that concentration of residue will not be as simple as hoped. One suggested alternative approach, which may be especially attractive for deep-pile fabrics, consists of collection by a vacuum method, followed by electrostatic precipitation in a small area. However, there does not appear to be any off-the-shelf equipment available to accomplish this, so that a considerable development effort would be required.

B. Dual Backscatter Detection.

The beam of electrons that is used to illuminate the specimen in the SEM creates the following three different signals that are of interest in this discussion: (1) Secondary electrons are dislodged from atoms in the specimen in a

very shallow volume element not significantly wider than the electron beam itself, (2) backscattered electrons are part of the incoming beam and are bounced back off atoms in a somewhat deeper and wider volume element of the specimen, and (3) x-rays, with energies characteristic of the elements present, are excited in a considerably deeper and wider volume element of the specimen.

Normally, the secondary electrons are used to form the image that is viewed. This is possible because the intensity of the secondaries is strongly modulated by surface detail, which is thus made visible, and also weakly by atomic number differences. This image has the best resolution because the secondary electrons come from the smallest volume. They are of low energy and are deflected toward the scintillation counter (which is off to the side) by a positively biased (typically 150 volts) grid placed over the detector.

The backscattered electrons, coming from a larger volume element, form an image with lesser resolution. However, being more sensitive to atomic number differences, their use is sometimes desired to selectively enhance the brightness of particles or areas containing heavier elements.

If the bias on the scintillation detector is removed, the secondary electrons will no longer be collected. Having higher energies and travelling in straight lines, the backscattered electrons can still reach the detector, but form a low intensity image because of the large angle between the beam axis and the direction to the detector. Furthermore, if in this mode the specimen is viewed at too shallow an angle to the surface, the backscattered electron image may not even show the looked-for atomic number effect and instead emphasize the topography of the specimen surface.

In order to make the best use of the backscattered electrons, a separate detector for them should be placed high in the specimen chamber, close to the incoming beam. Generally, a solid state detector with its own separate amplifier is used. This is an extra-cost option that is not available on all instruments. From the use of the single solid state detector, it is only a small step to the use of two of them, placed symmetrically around the beam.

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At the present time, two manufacturers will supply this arrangement, and more are expected to follow suit. For reasons that are still only imperfectly understood, the addition of the signals from the two solid state detectors very strongly enhances the sensitivity of the compound signal to differences in atomic number. With proper adjustment of the working distance, one can select a range of atomic numbers, such that elements with lower numbers (lighter elements) will practically fade from view, while elements with higher numbers (heavier elements) will stand out brightly. If the signals are subtracted instead of added, all atomic number effects are wiped out, and a topographic image is obtained which shows surface detail with excellent definition.

It was thought that dual backscatter detection in the signal addition mode might significantly speed the search for gunshot residue particles. The numerous particles containing only light elements would not be seen or seen dimly enough to be readily ignored, while particles containing lead, barium, and antimony (atomic numbers 82, 56, and 51) would stand out so brightly that they would be spotted at once. This was the case when the image was displayed on the normal viewing scope. However, the signal characteristics of the dual backscatter interface amplifier were incompatible with the TV monitor, and the image could not be viewed on the latter, which is essential for the rapid search procedure described in this report. The manufacturer has indicated that a TV-compatible interface could be designed and built at modest cost if there were a demand for it.

C. <u>Automation</u>.

Experience has shown that an efficient operator can complete two average gunshot residue cases, each involving several samples, in one working day. This is probably acceptable to many laboratories serving a limited area, but

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may impose an excessive burden on some large service laboratories. Automation may provide an answer to this problem.^a

Automation of laboratory experiments is no longer a novelty, and it is clear that the project entails the design of a small special-purpose computer or the appropriate programming of a small programmable computer to operate the instrument and to make some of the decisions that are now made by the human operator.

The first step in automating particle analysis is both obvious and trivial. It consists of motorizing the x and y stage motion controls, so that the computer rather than the human operator, can move them to examine picture after picture. The second step is to provide a mechanism for interrupting the search when a likely particle is encountered. This decision-making does not have an immediately obvious solution. In the present operator-performed procedure, visual recognition by morphology, aided by brightness, is used. The nature and range of criteria used by the operator may be impossible to program on a small computer. The operator's procedure could be reversed and an x-ray analysis could be done first. The results of this could easily be used by a computer. However, this requires an x-ray analysis for every particle, which is time-consuming and would therefore defeat the purpose of automation.

The use of the dual backscatter technique (see X. B) can provide a solution to this problem. If only particles containing heavy elements are bright, computer recognition of a likely candidate particle could be based simply and easily on a brightness criterion. With this hurdle cleared, the remainder of

^aDr. V.R. Matricardi of the FBI Laboratory has been a leading exponent of the concept of automating particle analysis. The proposals developed in this report have been arrived at independently, but were stimulated by his stand on this issue.

the automation project involves only engineering problems, and the entire procedure apparently consists of the following steps:

- Upon encountering a particle brighter than a certain preset level, searching is interrupted and magnification is increased, while the particle is kept centered. These are all routinely programmable steps once the computer is mechanically or electrically interfaced with the SEM controls.
- An x-ray analysis is performed, and the particle is automatically photographed if a permanent record is desired. This is not necessarily the case, but if it is desired, the photograph should be on a continuous film (micro or regular size) that can be automatically advanced after each frame.
- An even better way to preserve the picture would be to record it on TV tape, using an appropriate interface. This is likely to be much faster, because the currently-used photographic technique requires a very slow picture scan.
- To record the results of the x-ray analysis, the simplest but not necessarily best procedure would be to photograph or record the spectrum on the next frame.
- Since x-ray spectra are displayed on a separate monitor, the photographic method would require two cameras and two separate strips of film.
- The magnetic tape method would require only one recorder and one tape, since signals from different inputs can be switched by the computer.

An automated system need not be based on the simple x-ray system described in this report. One of the more sophisticated, computer-based systems could be used, which can evaluate the spectrum and either print or display the elements present by name for photographing or recording, superimposed on the picture of the particle, if desired. The final steps will depend on what the courts will accept and what the laboratory perceives to be its record-keeping requirements. The alternatives are: (a) to have the computer print a list of particles and their compositions, only a summary of this information, or both, or (b) to have the criminalist base his conclusions and testimony on his inspection of the film or his viewing of the tape.

When an automated system has been built, it may not resemble the one described, but the foregoing example indicates that there are no problems that are drastically different from problems that have already been solved in other applications. An automated system could probably be implemented at a cost that is reasonable for a laboratory with the work load that prompted these considerations.



CHAPTER XI. CONCLUSION

Particle analysis has been fashioned into the most definitive method of identifying and the most successful method of detecting gunshot residue to date. It identifies gunshot residue with greater certainty than any previous method because discrimination from a majority of occupationally caused deposits of lead, barium, or antimony is possible. It is effective for a much longer period of time after a firing than previous methods because particle analysis does not have a threshold problem. Analysis of about 120 actual cases has established the field readiness of this new method.

Gunshot residue can be found on a person's hand not only if the subject fired a gun, but also if he handled a recently fired gun or was a close bystander at a shooting. This investigation has contributed to clarification of the circumstances under which transfer of residue can occur and the amounts that can be expected as a result of such a transfer.

Finding particles other than gunshot residue particles can furnish forensically significant clues to the subject's recent activities or environment, both in firearms-related and other types of cases. Therefore, it would be useful to expand the range of environmental and occupational particles to be investigated.

The most important step that is now required is to increase the availability of the equipment and the skills to crime laboratories to carry out particle analysis. Beyond this, large service laboratories may require automation of the procedures. This is currently being considered at the FBI Laboratory.

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APPENDIX A. COMPOUNDS IDENTIFIED IN GUNSHO" RESIDUE PARTICLES

Like x-ray fluorescence, x-ray analysis in the SEM identifies the chemical elements that are present, but not the specific compounds into which they are combined.

Normally, the unique identification of specific solids is most readily accomplished by diffraction methods. There is probably too little gunshot residue on the hand, and at present it is too difficult to gather it all together, to produce a sample suitable for conventional x-ray diffraction (XRD) methods. It has been possible, however, to collect enough material from firings into plastic bags to obtain diffraction patterns that identified some of the ingredients.

Some of the residue collected in this fashion was also examined in a transmission electron microscope (TEM). This is not a suitable instrument for the search and particle analysis procedures described in this report, but it was used because it has an attachment that allows high-energy electron diffraction (HEED) patterns to be taken. The electron beam was not able to penetrate the micron-sized spheres, but it passed through the various protrusions (flakes, scales, spheroidal knobs) around the edges of the spheres' outline image. One or the other and sometimes both of these procedures identified the following materials:

8	Barium meta-antimonate	BaSb ₂ O ₆		
٠	Lead oxy-sulfate (Lanarkite)	$PbO \cdot PbSO_4$		
•	Graphite	С		
•	Lead	Pb		
•	Lead sulfide	PbS		
•	Antimony oxide (uncertain)	Sb ₂ O ₄		

These were limited experiments, and the list is undoubtedly incomplete.

It was also observed in the TEM that lead spheres in the direct electron beam often broke up at once into many smaller spheres. Since the beam intensity in the TEM is much higher than in the SEM, it was assumed that this was caused by melting of metallic lead, which has a very low melting point $(327^{\circ}C)$. In the SEM, occasional damage to lead particles was observed if they were very smal. Once formed, some of the new smaller spheres were seen to crystallize. The entire process of break-up to the crystallization of some of them took about three minutes under a beam in excess of 60 kV (up to 100). The electron diffraction patterns also changed in some cases, Lead spheres from a .32 caliber ACP cartridge (Llama pistol) changed to PbS for some and to a possible intermetallic compound BaPb₃ for others, presumably through a reaction with other materials that were present. Lead spheres from a .38 caliber special Remington 158 gr RNL cartridge gave initial patterns for PbO which changed to lead or to a mixture of lead and Pb₃O₄. An x-ray diffraction pattern of this residue revealed Pb, BaS, and PbO₂, with additional unidentified lines indicating be presence of still more compounds.

APPENDIX B. COMPUTER PROGRAM

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The table of probabilities for finding the particles described in Chapter VIII was calculated with the aid of a small computer program that can be readily altered to accommodate different geometries. A listing of this program is shown on the following page.

PR	OGRAM	AREA	76/76	OPT=1		FTN 4.6+4288	07/27/77	15.28.06
		PRC	GRAM AREA	(INPUT, OU	JTPUT, TAI	PE5=INPUT,TAPE6=OU	TPUT)	
		DINE	ENSION NC	(6), PROB (5	5,6)			
		DAT	A (NC(I), I	[=1,6) /1,	10,20,3	3,43,50/		
			= 131					
			= FLOAT(NT					
		COMMENT	NC = C =	NUMBER C	F CCLUM	NS SEARCHED.		
		CCMMENT	NT = T =	= TOTAL CO	LUMNS OF	N DISK.		
		COMMENT	NE = E =	NUMBER C	DF EMPTY	COLUMNS, EQUAL D1	STRIBUTION ASS	SUMEC
		COMMENT		FOR PART	ICLES,	WHOSE TOTAL IS SET	PROGRESSIVEL	Y AS
		COMMENT		5,10,15,	23,25 II	N THE FIRST LCOP T	HAT FCLLOWS.	
			1 I =1, 5					
			= 5*I					
			= NT-NP					
•			= FLOAT (NE	E)				
			= E/T					
			2 J=1,6					
			= NC(J)					
			J-1) 4,3,4	F				
			TO 2					
			5 K=2,IC					
			= K-1					
			= PR0 * 0		(T-CK)			
			B(I,J) = F	RO				
			TINLE					
			TE(6,6) (
						6X,1H5,8X,2H10,8X,		
			•		•	,5X,5(E10.3)/13		
			-			3X,2H30,5X,5(E10.3)/13X,2H40	•5X •
			iJ.3 ///	L3X,2H50,5	5X,5(E10	.3))		
		CAL	LEXIT					
		END						

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. 30

APPENDIX C. SEM OPERATING COSTS

The water and power needs of the SEM are modest. A constant supply of liquid nitrogen (LN) is needed to cool the x-ray detector crystal, which should be kept at liquid nitrogen temperature at all times. Allowing it to warm up to room temperature is detrimental and will ruin the detector crystal if this occurs too often. Operating it at room temperature will also ruin the detector crystal.

The electron gun filaments have a limited lifetime. In this project, the first several batches of filaments averaged 25 operating hours and the latest batch averaged 50 hours per filament. The reason for the difference is not known. The instrument came with a supply of ten filaments in a sturdy protective box that is used for both storage and shipping. It is advisable to purchase a second complete box of ten filaments. When the first ten are burned out, the assemblies can be sent out for rebuilding while the contents of the second box are being used. In this way, a supply of spare filaments is always on hand. The cost was \$72 for rebuilding ten of the filaments for the instrument used in this project. These filaments are made of tungsten. Some instruments use a different material (lanthanum hexaboride).

The SEM is under warranty the first year. During the second and subsequent years, a reasonable budget for service is at least \$1000. Normal downtime for one of the more reliable instruments is 5 percent, with part of it for routine cleaning of the column and apertures, and part for repairs.

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APPENDIX D. PARTICLE ATLAS

This appendix contains 76 photographs of gunshot residue particles, 16 photographs of flakes of smokeless powder found in residue, and 28 photographs of relevant environmental and occupational particles. This selection is representative of the varied appearances of gunshot residue particles and of similar particles that are generally encountered.

The legends beneath the photographs usually consist of three lines. The first line gives the diameter or the maximum and minimum dimension of the particle in micrometers. If the particle is too irregular to be measured, this number is omitted and a scale bar is placed on the picture. The next entries on the same line list the elements detected, in the order of their principal peak heights. Trace amounts are placed inside parentheses, but the distinction between minor ingredients and traces is not firm. In the case of the smokeless powder flakes, the major components are organic, but low-intensity spectra for inorganic elements are often obtained. At higher magnifications, these are generally seen to emanate from particle inclusions.

For gunshot residue particles, the second and third lines give the gun and the ammunition from which this particle was obtained. This should not be interpreted to mean that the particle is in any way characteristic of that combination. A sample of residue taken as a whole may have distributions of sizes, shapes, and compositions that are more characteristic of one ammunition than of another, but no single particle will convey such information.

Entries for the gun on Line 2 are listed as: the caliber, the make (also the model, if known), the barrel length in inches, and the type of cartridge for which the gun is chambered. The third line that identifies the ammunition does not list any information (such as caliber) that is already implied by the gun. The entries are: manufacturer, brand or series if required, weight of bullet in grains, and style of bullet.

For environmental and occupational particles, appropriate remarks are substituted for the second and third lines.

GUNSHOT RESIDUE PARTICLES



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1.8μm, Pb, Cu .357 Smith & Wesson, 6 in. Magnum W-W, 158 gr SWC



2.4 μ m, Ba, Si, Ca, K, Cu, Al, S, Cl .380 Browning Standard, ACP Rem, 95 gr FMJ



(b) 1.0µm, Pb, Cu, Sb .38 Charter Arms, 2 in., Special W-W, 158 gr RNL Lubaloy



(d) 5.0µm, Ba, Si, Ca .357 Smith & Wesson, 6 in., Magnum Norma, 158 gr SPFN



3.6µm, Ba, Ca, Si (Fe) 9 mm, Unknown Make S&W, 115 gr SJHP



6.4μm, Ba (Pb) 9 mm Browning Hi-Power W-W, 100 gr JHP



2.5µm, Ba, Si, Ca, Pb (K, Fe) 9 mm, Unknown Make S&W, 115 gr SJHP



2.5µm, Pb, Ba, Ca (Fe) .32, Rohm, RG..., 2 in., S&W W-W, 85 gr RNL











(b) 3.8µm, Pb, Sb, Ba, Fe, K, Cu .43 Colt, Gold Cup, ACP Winchester, 230 gr FMJ







(a) 20µm, Ca, Si, Ba .38 Smith & Wesson, 4 in., Special Rem, 125 gr SHJP



(c) 50µm, Ca, Ba, Si, S .32 Llama, ACP Browning, 71 gr FMJ



(b) 45µm, Ba, Ca, Si (Fe, S) .38 Smith & Wesson, 4 in., Special Rem, 158 gr FMJ





75 x 45μm, Ba, Ca, Si (Fe, Cu, Pb) 9 mm, Browning Hi-Power Fed, 123 gr FMJ

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(a) 52µm, Ca, Si, Ba . 380 Browning Standard, ACP Rem, 95 gr FMJ



(c) 14µm, Ba, Pb, Sb .38 Smith & Wesson, 2 in., Special Rem, 158 gr FMJ



(b) 40µm, Ba, Sb, Pb 9 mm, Unknown Make S&W, 115 gr SJHP



(d) 17µm, Pb, Sb, Ba . 380 Browning Standard, ACP Super Vel, 88 gr JHP

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6.0μm, Ba, Al, Sb, S .38 Smith & Wesson, 2 in., Special Rem, 158 gr FMJ



(b) 18µm, Ba, Sb, S . 380 Browning Standard, ACP Super Vel, 88 gr JHP



 $4.4\,\mu\text{m},$ Ba, Si, Ca, K, Al, Pb, P, Fe .38 Smith & Wesson, 4 in., Special Rem, 158 gr FMJ



(a) 15µm, Ba, Ca, Si, Pb, Fe . 380 Beretta, ACP Fed, 95 gr FMJ







60μm, Ba, Pb, Sb, Ca, Si . 357 Smith & Wesson, 2 in., Magnum Norma, 158 gr SPFN



24µm, Pb .38 Smith & Wesson, 2 in., Special Rem, 158 gr FMJ



(a) 38 x 28 µm, Ba, Ca, Si . 357 Smith & Wesson, 6 in., Magnum Super Vel, 110 gr J SP (SJFN)



(c) 20µm, Ca, Si, Ba, Pb (Fe) . 380 Beretta, ACP Fed, 95 gr FMJ



(b) 45µm, Ba (Pb) . 38 Smith & Wesson, 4 in., Special Rem, 158 gr FMJ



(d) 50µm, Si, Ba, Pb . 22 Colt, 6 in., LR We**s**tern, 40 gr Lubaloy

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(b) 40µm, Ba, Pb, Sb, AI .357 Smith & Wesson, 6 in., Magnum W-W, 158 gr SWC



(c) 28 x 16µm, Ba, Pb, Sb, Ca, Si . 357 Smith & Wesson, 6 in., Magnum Norma, 158 gr SPFN



50 x 25µm, Ba, Pb, Si, Ca, K, P, Al, Fe . 38 Smith & Wesson, 4 in., Special Rem, 158 gr FMJ



(a) 34µm, Si, Al, K, Pb, Ba, Fe .38 Smith & We**s**son, 4 in., Special Rem, 158 gr RNL



(c) 22 x 18µm, Pb, Cu . 22 Colt, 6 in., LR Western, 40 gr Lubaloy



(b) 20µm, Si, Al, K, Ba, Ca, Fe, Pb .38 Smith & We**ss**on, 4 in., Special Rem, 158 gr RNL



 $12\,\mu\text{m},$ Ba, Si, Ca, Pb, K (Fe, AI, P) . 38 Smith & Wesson, 4 in., Special Rem, 158 gr FMJ



(a) Ba, Pb, Sb, Al (Cu) . 357 Smith & Wesson, 6 in., Magnum W-W, 158 gr SWC



(C)

Ba, Ca, Si, Pb, K, Fe, Cu, Zn 9 mm Browning, Hi-Power Fed, 123 gr FMJ



(b) Ba, Ca, Pb, Si (Fe) . 380 Beretta, ACP Fed, 95 gr FMJ



Ba, Ca, Si, Pb, Cu, Fe, Al, K, Zn 9 mm Browning, Hi-Power Speer, 100 gr JHP



Browning, 71 gr FMJ



Ba, Ca, Pb, Si, Fe . 380 Browning Standard Rem, 95 gr FMJ



Ca, Ba, Si (Pb) .38 Smith & Wesson, 2 in., Special Rem, 158 gr FMJ



Ba, Ca, Si, Pb, K, Cu . 32 Llama, ACP Browning, 71 gr FMJ

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(a) 140 x 80 m, Cu, Ba, Pb, Zn 9 mm Browning, Hi-Power Speer, 100 gr JHP



(c) Ba, Pb, Ca, Si 9 mm Browning Hi-Power Fed, 123 gr FMJ



(b) Ba, Ca, Si (Pb) . 380 Browning Standard, ACP Rem, 95 gr FMJ



(d) Ba, Sb, Pb . 45 Colt Gold Cup, ACP Winchester, 250 gr FMJ



(a) Ba, Sb, Pb 9 mm Browning Hi-Power Speer, 100 gr JHP



(c) Ba (Pb, Ca, Si, Cu) . 32 Llama, ACP Browning, 71 gr FMJ



(b) Ba, Ca, Si (Pb) . 380 Browning Standard, ACP Rem, 95 gr FMJ



Ba, Pb, Sb 9 mm Browning Hi-Power W-W, 100 gr JHP



(a) 100µm, Ba, Si, Ca, K, Al, Fe (Pb, Cl, Cu) . 38 Smith & Wesson, 4 in., Special

Rem, 125 gr SJHP



(b) 150 x 50µm, Cu, Pb . 357 Smith & Wesson, 6 in., Magnum W-W, 158 gr SWC



Si, K, Al, Ba, Pb, Ca, Fe, P . 38 Smith & Wesson, 4 in., Special Rem, 158 gr FMJ



(c)

40μm, Pb .38 Smith & Wesson, 4 in., Special Rem, 158 gr FMJ


70μm, Pb, Si, Ca (Fe, Cu) . 22 Rohm Rosco, 2-1/2 in., Short Rem, 29 gr Brass Plated "Golden"



Pb, Cu, Ba . 22 Colt, 6 in., LR Western, 40 gr Lubaloy



(b) Pb, Cu . 22 Rohm Rosco, 2-1/2 in. Short Rem, 29 gr Brass Plated ''Golden''



(d) Pb, Cu . 22 Colt, 6 in., LR Western, 40 gr Lubaloy

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(a) 28µm, Pb, Cu . 22 Colt, 6 in., LR Western, 40 gr Lubaloy



(c) 12 x 10µm, Pb . 22 Colt, 6 in., LR Western, 40 gr Lubaloy



(b) 5.0 x 3.0µm, Ba, Pb, Cu (Ca, Fe) .22 Colt, 6 in., LR Western, 40 gr Lubaloy



20 x 12 μ m, Ba, Pb, Sb, Cl . 22 Colt, 6 in. , LR Fed Powerflite, 40 gr RNL

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CONTINUED 20F3









20µm, Pb . 22 Winchester 67A Rifle, 20 in., LR Sears, 40 gr RNL



(b) 14 x 11 µm, Pb (Sb) 9 mm, Unknown Make S & W, 115 gr SJHP



17 x 11 μ m, Ba, Si, Ca, Pb (K, Fe) . 32 Harrington & Richards, 2-1/2 in., S & W Rem 85 gr RNL



(a) 120µm, Powder Flake (Pb) . 22 Colt, 6 in., LR Fed Powerflite, 40 gr RNL



(c)

100μm, Powder Flake, [Ba, S, Sb, Cu, Fe, K, Si (Al, Pb)]
. 380 Browning Standard, ACP
Super Vel, 88 gr JHP



(b) 270 x 70µm, Powder Flake . 32 Llama, ACP Browning, 71 gr FMJ



220µm, Powder Flake (Ba, Ca, Si, Pb) . 380 Browning Standard, ACP Rem, 95 gr FMJ



(a) 160 x 30 µm, Powder Flake (Ba, Pb, Ca, K, Cu) . 380 Browning Standard, ACP Super Vel, 88 gr JHP



(c) Powder Flake (Ba, Sb, Pb) 9 mm Browning Hi-Power W-W, 100 gr JHP



. 370 Browning Standard, ACP Rem, 95 gr FMJ



210µm, Powder Flake 9 mm Browning Hi-Power Speer, 100 gr JHP

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(a) 100µm, Powder Flake (Pb) 9 mm Browning Hi-Power Fed, 123 gr FMJ



230 x 52µm, Powder Flake (Pb, K) . 38 Smith & Wesson, 2 in., Special Rem, 158 gr FMJ



Powder Flake (Ba, Sb, Ca, S, Si) .38 Smith & Wesson, 2 in., Special Rem, 158 gr FMJ



100μm, Powder Flake .38 Smith & Wesson, 4 in., Special Rem, 125 gr SJHP





. 357 Smith & Wesson, 6 in., Magnum Norma, 158 gr SPFN



(C)

375 x 75μm, Powder Flake, [Pb (Cu)] .357 Smith & Wesson, 6 in., Magnum W-W, 158 gr SWC



(b) 175µm, Powder Flake (Pb, Sb, Ba) .357 Smith & Wesson, 6 in., Magnum W-W, 158 gr SWC



380µm, Powder Flake . 44 Hawes, 6 in., Magnum W-W, 240 gr(?) Rem-Mag

ENVIRONMENTAL AND OCCUPATIONAL PARTICLES



(a) 3.0µm, Ce, La Common From Lighter Flints



(c) 11µm, Fe Common



7.5 μ m, Ce, La, Fe Common From Lighter Flints





(Ba SO4 and SO₄ are widely distributed as pigments, opacifiers, thickeners, extenders, & paper coatings in many commercial products)







6.4µm, Ti Common

(TiO₂ is widely used in paint pigments and cosmetics)



(c) 1.8µm, Pb, Cl, Br Automobile Exhaust



(d) 5.0 x 4.0µm, Pb, Cl, Br Automobile Exhaust



(a) 25µm, Ba, Si, Pb, K, Ca, Fe . 22 Low Velocity Studgun Omark Cartridge



3.5µm, Si, Ba, K, Ca, Pb (Cl, Fe, Zn) . 22 Low Velocity Studgun Omark Cartridge



(b) 3.6µm, Ba, Pb (K, Ca) . 22 Low Velocity Studgun Omark Cartridge



3.2μm, Ba, Pb, Fe, K, Ca, Zn, Cu .22 Low Velocity Studgun Omark Cartridge







(b) 3.0µm, Pb(Zn) Lead Smelter



(c) 5.0µm, Pb(Sb, Cu, Zn) Lead Smelter



(d) 3.6µm, Pb, Zn Lead Smelter



(a) 3.5µm, Pb, Cu Lead Smelter



(b) 3.5 x 2.5µm, Sb, Pb, Fe, Zn Lead Smelter



(c) 2.5 x 2.0 μ m, Pb, Sb, Cu Lead Smelter



(d) 3.2µm, Pb, Sb, Cu Lead Smelter



(a) 1.2µm, Pb, Sb Lead Smelter



(b) 22 x 19 µm, Pb, Sb Lead Smelter



(c) 11 μ m, Pb (Sb, Cu, Zn, Fe) Lead Smelter



(d) 64 μ m, Ti, Si, Fe, Mn Welder



2.8 μ m, Pb Automobile Brake Mechanic



3.6 μ m, Pb, P, Ca, Ba, Zn, Cl, Fe Automobile Brake Mechanic



(c) 33 μ m, Pb, Fe, Cu, Si, Ba, Cl Automobile Brake Mechanic



(d) 18 x 13µm, Cu, Zn, Pb Machini**s**t



APPENDIX E. SAMPLE CASE REPORTS TO REQUESTING AGENCIES

This appendix comprises four representative case reports. The names of agencies and subjects have been deleted.

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Post Office Box 92957, Los Angeles, California 90009, Telephone: (213) 648-5000

REPORT OF EXAMINATION FOR GUNSHOT RESIDUE BY PARTICLE ANALYSIS (Page 1 of 5). (Scanning Electron Microscopy with Qualitative Elemental X-Ray Mentification)

SUBMITTING AGENCY	<u>Aura-Turren kilonii Saanta (</u> S	and the second
ATTENTION	Werther Kuliger	DATE OF REPORT 5/27/77
AGENCY'S CASE ID	ageneration and a second second	
AEROSPACE CASE NO.	158	

LIST OF SAMPLES EXAMINED, FRACTION OF AREA SCANNED FOR EACH, DATES OF EXAMINATIONS:

Item 1.)	Standard lift sample	0.50% Area Scanned	5/24/77
Item 2.)	Right Web, back sample (B-2)	1.50% Area Scanned	5/24/77
Item 3.)	Left web, back sample (B-3)	1.50% Area Scanned	5/24/77

RESULTS OF EXAMINATION(S):

(If residue is found, lists of particles will be attached)

Item 1.) Gunshot residue was found.

Item 2.) Gunshot residue was found.

Item 3.) Gunshot residue was found.

ANALYST:	R. S. Meslitt	61.177
SUPERVISOR:	R. S. Nesbitt	
-	G. M. Wolten	

COMMENTS AND INTERPRETATIONS, IF ANY, WILL BE GIVEN ON PAGE 2. COPY OF CASE FACT SHEET IS ATTACHED.

An Equal Opportanity Imployer CLNERAL OFFICES LOCATED AT: 2350 LAST CL SEGUNDO BUULEVARD KL SEGUNDO, CALIFORNIA

REVISED 12/2/76

PLEASE USE ONE FORM FOR EACH SUSPECT

THE AEROSPACE CORPORATION

GUNSHOT RESIDUE FROGRAM

DATA SHEET FOR GUNSHOT RESIDUE SPECIMENS, SUBMITTED FOR SCANNING ELECTRON MICROSCOPY WITH X-RAY ELEMENTAL ANALYSIS.

SUBMITTING AGENCY SANTA	
SUBMITTED BY	PHONE DATE 5/24/77
CASE NUMBER CHE	CK HERE ONLY IF SUICIDE
SUSPECT'S NAME OR ID .	5EXM
	TY PRIOR Purchasing gasolene
HOURS BETWEEN FIRING AND SAMPLING 17	UTDOORS NO. SHOTS 3
ACTIVITY BETWEEN FIRING AND SAMPLING TO hosp	ital by ambulance DOA, to mortuary
DID SUSPECT WASH HANDS BEFORE SAMPLING ?	VES MAYBE NO
WEAPON TYPE Revolver CALIBER . 38 Spec.M/	
AMMO BRAND/TYPE, DESCRIBE AS FULLY AS POSS.	
BULLET TYPE . <u>38 Special</u> BULLET WEIG (only 2 of 3 shots recovered)	HT <u>125</u> Jacketed Scmijack., Hollow Point Plated
	BARE LEAD
SAMPLE IDENTIFICATIONS (LABELS)	
RIGHT WEB, BACK B-2 , RIGHT WRIST	, RIGHT SLEEVE
LEFT WEB, BACK B-3 , LEFT WRIST	
LIP OF INSIDE OF RIGHT PANT'S POCKET	, LEFT
DESCRIBE ADDITIONAL SAMPLES ON BACK OF THIS IN A NON-STANDARD MANNER ON THE BACK OF THIS	
IT IS REQUESTED THAT A TEST-FIRING HANDSAMPLI And Ammunition involved, if Available.	E BE SUBMITTED, USING THE WEAPON
WAS WEAPON TEST-FIRED ? Yes	IF YES, DESCRIBE TEST-FIRING
(HORIZONTAL/VERTICAL, INDOOR/OUTDOOR, ETG.)	
hand back and web fired with suspects R-P s	
semi-jacketed S. P.	
AEROSPACE CASE NUMBER 158	THE IVAN A. GETTING LABORATORIES THE AEROSPACE CORPORATION Box 92957, Los Angeles, CA 90009
REMARKS: Suspect was shot by an officer from approximately 60 feet.	G.M. WOLTEN (213) (48-6944 AND STAFF.
-pp-shinadon, of asses	LOCATION OF LABORATORIES:

AEROSPA CASE NO D.5	. 158 0% Area Scanned	SAMPLE <u>Stundard - lift</u>	•	(THIS
TYPE	SIZE	MAJOR ELEMENTS	MINOR EL	EMENTS
*	(Micrometers)	**	**	Traces
S	1.5	Pb		55
222	0.8	Pb		SP.
S	1.5	Pb		Sb
. <u>Š</u>	1.8	Pb		Sь
- 1	1,0	Pb		36
1	7.5	Pb		.•
S S	.4.8	Pb		Ba Cu Si
S	4.2	РЪ		i i
S	2.1	Pb		Sb Ва
1	3.8 × 1.3	Pb Ba	S6K	
.2.	1.7	Pb		56
<u>S</u>	1.6	Pb		
2 2 2 2 2 2 2	2.5	Ba	P6 S. Ca	K Fe
S .	1.0	Pb		SP
S	4.2	Ba Pb	(a K	1
S	1.5	Pb Ba	GK	Cu
1	5,8	Pb Ba	Cak	
l [*]	2.9	Pb ·	Ba Sb	
1	3.8	РЬ		SЬ
S	0.8	PPP		
5555	1.8	Pb		Sb Br
S	2.0	Pb		56
S	1.3	Pb		
	2.0	Pb	Ca	-
1	2.5	.Pb		Ba Sb
S	0. <i>5</i>	PL	a la ser en el ser	
S S	<i>i</i> .7	Pb		
S	5.0	Ba	Gas;	KAISFEC
S S	1.6	Pb		
S	3.3	Ba Pb	SKK	Cu
	÷			

PARTICLE LIST

<u>/.5%</u> TYPE	SIZE	MAJOR ELEMENTS	MINOR ELEME	NTS
*	(MICROMETERS)	**	**	Traces
S	1.8	Sb		
	.2.1	РЬ		SP.
S S S	1.7	Pb		
S	1.7	Pb	Sb	Cu
	4.6	Pb		12P
S	2.5	Pb		K (a
S S	1.7	Bas. Ca		FESK Ph
<u>_S</u>	4.2	РЪ		56
1	2.0 ×1.0	Pb		1
S	5.0	PL.	Ba Sb	
1	2.1	Рb		Sь
<u> </u>	1.9	РЬ		SЬ
S S	1.5	Pb		
S	2,1	Ba Si Ca	Æ	36
1 -	6.7	РЬ		פר
<u></u>	2.5	Pb		
S	1.7	РЬ		
S	1.3	РЬ	Sb Ba	
5 5 5 5 5 5 5 5	0.8	Pb		Sh Ba K Fi
3	2.3	РЬ		+
S	1.3	Pb	SP	SР
S	0.8	Pb		
S	0.5	Pb	St	SP
	3.3	Pb Ba	Sb	
2 S	1.7	Ba Pb	CIGK ShS.	Fe
	0.8	Pb	Ba Sb	
2	1.3	РЬ РЬ		56 56
<u></u>	1.2	<u>Pb</u>	-	56
5				

** ELEMENTS WITH ATOMIC NUMBERS BELOW 12 (Mg) ARE NOT DETECTED (E.G. NA, C).

-177-

	. <u>158</u> 5 <u>Area Sanned</u> SIZE	AMPLE Left web back	•	
TYPE *	SIZE (Micrometers)	MAJOR ELEMENTS	MINOR ELEM	ENTS Trace
S	2.1×1.3	РЬ		56
S 1 2 5	3.8	Pb Ba	Sь	K
S	2.5	Pb		
	2.9	РЬ		156
1	3.3	Ba	PBS K Sb	Al Fe
2	2.9	РЬ Ва РЬ	Cas, SbK	SР
1 2 2 2		Pb	Ba Sb	
<u> </u>	<u></u>		154 00	
**********		• • •		
	4.			
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REPORT OF EXAMINATION FOR GUNSHOT RESIDUE BY PARTICLE ANALYSIS (Page 1 of 5). (Scanning Electron Microscopy with Qualitative Elemental X-Ray Identification)

 SUBMITTING AGENCY

 ATTENTION

 Junk

 DATE OF REPORT 5/27/77

ATTENTION DATE OF REPORT 5/27/ AGENCY'S CASE ID

LIST OF SAMPLES EXAMINED, FRACTION OF AREA SCANNED FOR EACH, DATES OF EXAMINATIONS:

Item 1.)	Standard Back Sample	1.5% Area Scanned	5/26/77
Item 2.)	Right hand Back/Wrist Sample	2.3% Area Scanned	5/26/77
Item 3.)	Right hand Wrist/Palm Sample	5.4% Area Scanned	5/26/77

RESULTS OF EXAMINATION(S):

(If residue is found, lists of particles will be attached)

Item 1.) Gunshot residue was found.

Item 2.) Particles consistent with being gunshot residue were found.

Item 3.) Gunshot residue was found.

ANALYST: 7	C.S. Mabett 01.177
SUPERVISOR:	R. S. Nesbitt
	G. M. Wolten

COMMENTS AND INTERPRETATIONS, IF ANY, WILL BE GIVEN ON PAGE 2. COPY OF CASE FACT SHEET IS ATTACHED.

AN Equal Opportunity Employer General Offices Located at. 2360 cast el begundo boulevariu, el begundo, california

PLEASE USE ONE FORM FOR EA	REVISED 12/2/76
THE AEROSPACE CORPORATION	GUNSHOT RESIDUE FROGRAM
DATA SHEET FOR GUNSHOT RESIDUE SPECIMENS, 5 Microscopy with X-Ray Elemental An	
SUBMITTING AGENCY	A CALLER AND A CALLER
SUBMITTED BY	ELEPHONE
CASE NUMBER CI	HECK HERE ONLY IF SUICIDE
SUSPECT'S NAME OR ID	SEX
HOURS BETWEEN FIRING AND SAMPLING approx	UNDOORS NO. SHOTS
ACTIVITY BETWEEN FIRING AND SAMPLING AFTER	
DID SUSPECT WASH HANDS BEFORE SAMPLING ?	YES MAYBE NO
WEAPON TYPE <u>Shotgun</u> CALIBER <u>12 gauge</u> AMMO BRAND/TYPE, DESCRIBE AS FULLY AS POS BULLET TYPE <u>No. 7-1/2 shot</u> BULLET W	s. Federal Hi-Power EIGHT <u>1-1/8 ounce</u> Jacketed Semijack.
	PLATED BARE LEAD Bare Lead
SAMPLE IDENTIFICATIONS (LABELS)	
RIGHT WEB, BACK A-11 , RIGHT WRIST / F	
LEFT WEB, BACK, LEFT WRIST	, LEFT SLEEVE
LIP OF INSIDE OF RIGHT PANT'S POCKET	, LEFT
DESCRIBE ADDITIONAL SAMPLES ON BACK OF THI IN A NON-STANDARD MANNER ON THE BACK OF TH	
IT IS REQUESTED THAT A TEST-FIRING HANDSAM AND AMMUNITION INVOLVED, IF AVAILABLE.	IPLE BE SUBMITTED, USING THE WEAPON
WAS WEAPON TEST-FIRED ? Yes	IF YES, DESCRIBE TEST-FIRING
(HORIZONTAL/VERTICAL, INDOOR/OUTDOOR, ETC.	
standard back from test firing 1 time	
AEROSPACE CASE NUMBER 160	THE IVAN A. GETTING LABORATORIES THE AEROSPACE CORPORATION Box 92957, Los Angeles, CA 90009
REMARKS:	G.M. WOLTEN (213) (48-6944 ANU STAFF.
	LOCATION OF LABORATORIES: 300 S. Douglas St., EL Segundo

-180-

Aerosp Case N <i>1.5</i>	0. <u>160.</u> SI	MPLE Standard Back	PAG (Test Fire)
TYPE	SIZE (MICROMETERS)	MAJOR ELEMENTS	MINOR ELEMENTS
*	(MICROMETERS)	**	## Trac
S	2.9	Pb	P Ca Sb Fe
S S	1.3	Pb	56
1	4.0	РЬ	
<u>`S</u> `	2.5	РЬ	CI SB
S	1.2	Pb	56
S	0.8	PЬ	56
S S S	1.3	Pb	Sb
	1.9	Ph Ba	CI SE C
S S	2.7	Pb	56
S	0.9	Pb CI	Sb Fe
a			- [
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|. LE)

TYPE	Size	MAJOR ELEMENTS	MINOR ELEM	ENTS
#	(MICROMETERS)	**	**	Traces
2	2.7	Pb	Sb Ca	T
2	1.0	Pb		SU
5 5 5 5	1.5		55	
<u>Š</u>	0.8	Pb Pb		
S	1.3	РЬ		56
S	1.7	P6	CI I	1
2 2 2 2	4.2	Pb		SЬ
	1,0	Pb		56
2	2,1	PL,		51
ļ	3.8	РЬ РЬ		56 56
2	6.7 3.8	Pb		56
1 2 2 2 2	1,3	Pb	······	Sb
2	1,9	Pb		Sb -
0				
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·····				
			: :	
			1	ī

PARTICLE LIST

PARTICLE LIST

TYPE *	Area Scanned SIZE (MICROMETERS)	MAJOR ELEMENT'S **	MINOR ELEMENTS
2 2 2	2.2 2.1 5.0 2.5	РЬ РЬ Ва РЬ	Fe S Sb
2 2 2 2 2 2 2 2 3	8.0 3.8 9.2 9.6	РЬ РЬ РІ, РЬ	CI

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REPORT OF EXAMINATION FOR GUNSHOT RESIDUE BY PARTICLE ANALYSIS (Page 1 of 5). (Scanning Electron Microscopy with Qualitative Elemental X-Ray Identification)

SUBMITTING AGENCY	Bon Brown Stan Banaillin Ollinge			
ATTENTION	ter in the terms	DATE OF	REPORT_6/23/77_	
AGENCY'S CASE ID	rizer Weiter			
AEROSPACE CASE NO.	170			

LIST OF SAMPLES EXAMINED, FRACTION OF AREA SCANNED FOR EACH, DATES OF EXAMINATIONS:

ltem 1.)	Test fire sample	10% Area Scanned	6/23/77
Item 2.)	Victim #1 right hand sample	10% Area Scanned	6/23/77
ltem 3.)	Victim #1 left hand sample	10% Area Scanned	6/23/77
ltem 4.)	Suspect (victim#2) right hand sample	10% Area Scanned	6/23/77
ltem 5.)	Suspect (victim #2) left hand sample	10% Area Scanned	6/23/77

RESULTS OF EXAMINATION(S):

(If residue is found, lists of particles will be attached)

- Item 1.) Gunshot residue was found.
- Item 2.) No gunshot residue was found.

THE AEROSPACE CORPORATION

Item 3.) No gunshot residue was found.

Item 4.) Gunshot residue was found.

Item 5.) Gunshot residue was found.

ANALYST: SUPERVISOR:

COMMENTS AND INTERPRETATIONS, IF ANY, WILL BE GIVEN ON PAGE 2. COPY OF CASE FACT SHEET IS ATTACHED.

An Equal Opportunity Employer general offices located at: 2330 east el secundo boulevard, el begundo, california

THE AEROSPACE CORPORATION	GUNSHOT RESIDUE FROGRA'I
DATA SHEET FOR GUNSHOT RESIDUE SPECIMENS, S Microscopy with X-Ray Elemental An	• · · · · · · · · · · · · · · · · · · ·
SUBMITTING AGENCY	and the second
SUBMITTED BY	ELEPHONE DATE _ 5/18/77
	HECK HERE ONLY IF SUICIDE
SUSPECT'S NAME OR ID	SEXM
OCCUPATION Retired ACT	VITY PRIOR Unknown
HOURS BETWEEN FIRING AND SAMPLING 16 to 1	B hours OUTDOORS
ACTIVITY BETWEEN FIRING AND SAMPLING Betwee	
DID SUSPECT WASH HANDS BEFORE SAMPLING ?	YES MAYBE NO
Semi-Automatic 9 mm WEAPON TYPE <u>Pistol</u> CALIBER Para	
AMMO BRAND/TYPE, DESCRIBE AS FULLY AS POSE 9 mm full jacket	S•
BULLET TYPE_exposed baseBULLET W	EIGHT 124 grain JACKETED SEMIJACK.
	PLATED BARE LEAD Jacketed
SAMPLE IDENTIFICATIONS (LABELS) Vict. Suspect	
RIGHT WEB, BACK A-8 A-9, RIGHT WRIST	, RIGHT SLEEVE
LEFT WEB, BACK A-8 A-9 , LEFT WRIST	, LEFT SLEEVE
LIP OF INSIDE OF RIGHT PANT'S POCKET	, LEFT,
DESCRIBE ADDITIONAL SAMPLES ON BACK OF THI IN A NON-STANDARD MANNER ON THE BACK OF TH	
IT IS REQUESTED THAY A TEST-FIRING HANDSAM AND AMMUNITION INVOLVED, IF AVAILABLE,	PLE BE SUBMITTED, USING THE WEAPON
WAS WEAPON TEST-FIRED ? Yes	IF YES, DESCRIBE TEST-FIRING
(HORIZONTAL/VERTICAL, INDOOR/OUTDOOR, ETC.	Right web, back of hand, after a
single test firing, box marked A-1 test.	
AEROSPACE CASE NUMBER 170	THE IVAN A. GETTING LABORATORIE THE AEROSPACE CORPORATION
REMARKS:	BOX 92957, LOS ANGELES, CA 9000 G.M. Wolten (213) 448-6944
*Murder-Suicide	AND STAFF.
	LOCATION OF LABORATORIES: 300 S. Douglas St., EL Segundo
	Deb St housing birth an easterne

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AEROSPAC Case No.		SAMPLE Test fire		(Tuis
TYPE *	SIZE (MICROMETERS)	MAJOR ELEMENT'	MINOR ELEME	TRACE
1	260 x 50	Ba Ca S	Pb Sb	
2	25	Ba Ca Si	2 62	Pb Fe
5 5 5	13	Ba S	S6 Pb	
2	21	Ba Ca Si	<u>56 S</u>	РЬ
S	21	Ba Si Ca		
S	11	Ba Ca Sb Si S		Pb
2 2	4.6	Ba Si Ca	K	Fe
	75 x 17	Ba Ca Si Pb	36	KCu
S	29	· Ba Gasi		
S S	12	Ba Ga Si		
S	32	Ba Ca	Si	
2	17	Ba Ca Si S	S6 P6	<u> </u>
1	33 x 17	Ba Ca	5: 56 5	IP6
S	27	Ba Ca. Si	56 S	Pb
1	108 × 58	Bas Gasi	P6 S6 K	
<u>s</u>	9.2	Pb		
S	54	Ba Ca	Siks	Fe
2	3.5	Ba Si Ca	1	
2 2 2 2	21	Ca Ba Si	S	
S	3,3	<u>i Cu</u>		Zn
2	42 × 29	Ba Ca	2 32:24	Pb
1	21	Pb		
S	27	Ba	Ca Si S Sh Fe	Pb
				-
	, in		· · · · · · · · · · · · · · · · · · ·	
			a a	ļ

-186-

Acrosp Case II	ACE 0. <u>170</u> S/	MPLE SHSpect (V#2) rig	out hand	PAGE / (THIS
түре *	SIZE (MICROMETERS)	MAJOR ELEMENTS	MINOR ELEMENT	TS
S I S	3.3 63 × 50 33 × 25	Ba S. Ca Ba Ca S: Br Ca S;	K Pb	K Fe P
		•		
		•		

-187-

AEROSPA CASE RO	s. <u> </u>	AMPLE <u>Suspect(V#2)</u>	left hand	PAGE / (THIS
TYPE *	SIZE (MICROMETERS)	MAJOR ELEMENTS **	MINOR ELEMENT	S TRACE
1	54	Pb		<u> </u>
C)	31	Ba Ca S.	Pb K Fe Cakci Fe Si S Cu	
1 S.	2.1	Pb		
<u>د</u>	2.9	Ba Ba Ca	<u>S;</u>	Fe K
S S	1.6	Pb	F"	PER
S ,	0.8	Cu		Zn
		an a search an	1	
	an al'al'an anna 1 de starras de a ranne d'anna an an an an		· · · · · · •	
				-
· · · · · · · · · · · · · · · · · · ·				
				ŀ
gas - Parlan alfred - region a arms		•		
· .				
a analasian darat ta pananaire				
-		•		
			•	
		LEGULAR, C = CLUSTER, F -		ł



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REPORT OF EXAMINATION FOR GUNSHOT RESIDUE BY PARTICLE ANALYSIS (Page 1 of 6). (Scanning Electron Microscopy with Qualitative Elemental X-Ray Identification)

CTTD > (TOURTNIC	A CONTRACTOR	South Martin Constant Constant
SURMITING	AGENCY	

ATTENTION	Mini Quintania	DATE OF REPORT 8/26/77
AGENCY'S CASE ID		
AEROSPACE CASE NO.	187	

LIST OF SAMPLES EXAMINED, FRACTION OF AREA SCANNED FOR EACH, DATES OF EXAMINATIONS:

Item 1.)	Right web, back sample	5% Area Scanned	8/20/77
Item 3.)	Left web, back sample	5% Area Scanned	8/20/77

RESULTS OF EXAMINATION(S): (If residue is found, lists of particles will be attached)

Item 1.) Particles typical of gunshot residue were found. Item 3.) Particles typical of gunshot residue were found.

ANALYST: R J. 8-30-77 110 R. 5/3417 SUPERVISOR: 飞机 G.^CM. Wolten

COMMENTS AND INTERPRETATIONS, IF ANY, WILL BE GIVEN ON PAGE 2. COPY OF CASE FACT SHEET IS ATTACHED.

An Equal Opportunity Employer SEMERAL OFFICES LOCATED AT: 2380 KABY EL BEGUHOO BOULEVARD, EL BEGUHOO, CALIFORMIA

Post Office Box 92937, Los Angeles, California 90009, Telephone: (213) 648-5000

REPORT OF EXAMINATION FOR GUNSHOT RESIDUE BY PARTICLE ANALYSIS (Page 1 of 6). (Scanning Electron Microscopy with Qualitative Elemental X-Ray Identification)

SUBMITTING AGENCY	<u>yaan aa saa kaasin </u>	
		DATE OF REPORT <u>8/26/77</u>
AGENCY'S CASE ID	28994-:	
AEROSPACE CASE NO.	187	

COMMENTS AND INTERPRETATIONS

Because of the rich deposits of particles typical of gunshot residue on Items 1 and 3, Items 2 and 4 (wrist samples) were not examined.

COMMENTS AND INTERPRETATIONS, IF ANY, WILL BE GIVEN ON PAGE 2. COPY OF CASE FACT SHEET IS ATTACHED.

> An Equal Opportunity Employer General Oppices located at. 2350 east el segundo boulevard, el degundo, california

Revised	12/	'2/	75
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PLEASE	USE	ÖNE	FORM	FOR	EACH	SUSPECT
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GUNSHOT RESIDUE FROGRAM

DATA SHEET FOR GUNSHOT RESIDUE SPECIMENS, SUBMITTED FOR SCANNING ELECTRON MICROSCOPY WITH X-RAY ELEMENTAL ANALYSIS.

SUBMITTING AGENCY
SUBMITTED BY
CASE NUMBERCHECK HERE ONLY IF SUICIDE_ Possible
SUSPECT'S NAME OR ID SEX M
OCCUPATION Used Car Salesman To SHOTLING Unknown
HOURS BETWEEN FIRING AND SAMPLING 20.5 INDORS NO. SHOTS X
ACTIVITY BETWEEN FIRING AND SAMPLING deceased
DID SUSPECT WASH HANDS BEFORE SAMPLING ? YES MAYBE NO
WEAPON TYPE Revolver CALIBER . 22 MAKE/MODEL OF BARREL
AMMO BRAND/TYPE, DESCRIBE AS FULLY AS POSS.
BULLET TYPE BULLET WEIGHT JACKETED
SLAIJACK. PLATED
BARE LEAD
SAMPLE IDENTIFICATIONS (LABELS)
RIGHT WEB, BACK, RIGHT WRIST, RIGHT SLEEVE
LEFT WEB, BACK 3 , LEFT WRIST 4 , LEFT SLEEVE
LIP OF INSIDE OF RIGHT PANT'S POCKET, LEFT

DESCRIBE ADDITIONAL SAMPLES ON BACK OF THIS FORM. DESCRIBE ANY SAMPLES TAKEN IN A NON-STANDARD MANNER ON THE BACK OF THIS FORM.

IT IS REQUESTED THAT A TEST-FIRING HANDSAMPLE DE SUBMITTED, USING THE VEAPON AND AMMUNITION INVOLVED, 1° AVAILABLE.

WAS WEAPON TEST-FIRED ? _____, IF YES, DESCRIBE TEST-FIRING (HOL JUNTAL/VERTICAL, HIDOGY/OUTDOOR, ETC.)

ALROSPACE CASE NUMBER 187 Tarrant County Sheriff's 0 Office Case RLMARKS: Gunshot wound to left forehead (subject was right-handed). Subject's hands were bagged after shooting in plastic bags; copious amounts of condensed moisture on hnads; air-dried before sampling. THE IVAN A. GETTING LABORATORIES THE AEPOSPACE CORPORATION Box 52957, Los Angeles, CA 93659

G.M. WOLTEN (213) (18-691*

LOCATION OF LABORATORIES: 300 S. DOUGLAS ST., LL SEGUNDO

1	AGLOSPI VADE RO		SAMPLE Right web, back			
	TYPE	SIZE (MICROAUTURS)	HAJOR ELEMENTS	MINGR ELEMENTS		
	S	31	Pb Cu	Zn		
	د ۲	38	Pb	Cu ···		
		13	Pb	ci cu		
, i	1	4.2	Pb			
ļ	2		Pb	CI Si Cu		
•	2	- 3.5		CI Cu		
	S	1.5	Pb Sici	Cu Ca		
		. 3.1	Pb Bl	CI Cu		
	<u></u>	7.3	<u>Pb</u>	<u>Cu</u>		
1	1	42	Pb Cu	Zn		
	5	6.9	Pb	CI Si Cu Ca		
- 1	S	1.3	Pb	Si CI Cu		
·		19	<u>Pb</u>	Cu Cl		
	1	4.0	Pb ·	Si CI Cu		
	S	2.7	Cu Zn.			
	1	12	Pb Cu	(1 Ga Zn		
.]		423 x 270	Pb Cu Fe	<u>Si Ci Ca Zn Ba</u>		
	S	· 31	Pb			
	S	1.9	Pb CI	Si Ca Cu		
1	1	2.3	Pb Cl	Ca Cu		
	1	40	Pb	Си		
	1	115	Pb Cu	Zn Ca Cl		
	1	27	Cu Pb Zn.	ci Ca		
	1	9.6	Pb Cu	CI Zh		
	i	115 × 46	Pb			
				Cu Zn Cl Cu		
		100 77 × 38	.Pb			
		2.1	Pb PL CL C	Cuizn Ca		
	,	1.9	Pb CI Si	Cu		
		and the second	<u></u>	<u>CI Cu</u>		
	2	9.6	Pb Di	Cu CI Ca P Zn		
	2	1.7	Pb Cu			

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* S & SPHEPOIDAL, I = IRRECULAR, C = CLUSTER, F = POWDER FLAKE. ** ELEMENTS WITH ATOMIC HUMBERS BELOW 12 (MG) AND ROT DETECTED (C.G. NA, L).

A i C	erospi Ase Ni	nce 	SAM	IPLE <u>Right web, back</u>		PAGE 2 0 (THIS S
T	YPE *	SIZE (MICROMETERS)		MAJOR ELEMENTS	MINOR ELEMEN	r
					<u> </u>	TRACE
-	S	1.2		Pb	Cu	
2		1.3		Pb cl (u	CICI	
	-	3.1		Pb		
5		1.7		Pb Cu		
5		1.0		Pb	CI Cu	
		2.3		РЬ	CI Si	
	5	2.7		S: Pb	CaCIK Fe CuZ	n
	5	1.5		Pb	CI Cu	
-	<u>د</u>	12		Pb		Gu
-	S	7.7		Pb	CI Cu	
-	222	1.7		Pb	CI Ca Cu	
	7	1.9		Pb	CI Sh Cy Zn	
					-	
						}
				GULAR, C = CLUSTER, Γ =		

PARTICLE LIST

AEROSPACE CASE No. 187

SAMPLE Left web. back

PAGE / OF 1. (This Sample)

TYPE *	SIZE (Micrometers)	MAJOR ELEMENTS	MINOR ELEMENTS	TRA CI
F	650 × 460	Pb Cu	Zn	CI Ca
1	54× 27	Pb	Cu Cl Zn	
S	8.6	РЬ		
	8.5 x 4.3	<u>Pb</u>	CI Ca	Cu
S	38	Pb		Си
	85 × 46	Pb St DL C		
1	19 × 12	Si Pb Ca Pb		
2	4.2	Pb Cu Cl	Zn	
S	4.4	РЬ		
1	27	РЬ		
	46×23	Pb	Cu	Zn
F	850	Pb Cu	P CI Ca	
1	23×13	Pb		
1	8.5 × 5.4 54	РЬ РЬ		
·]	12	Pb		
	a			
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NOTES

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