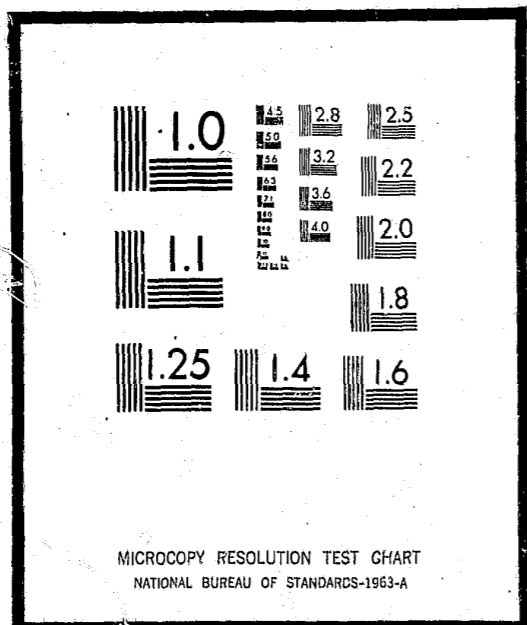


NCJRS

This microfiche was produced from documents received for inclusion in the NCJRS data base. Since NCJRS cannot exercise control over the physical condition of the documents submitted, the individual frame quality will vary. The resolution chart on this frame may be used to evaluate the document quality.



Microfilming procedures used to create this fiche comply with the standards set forth in 41CFR 101-11.504

Points of view or opinions stated in this document are those of the author(s) and do not represent the official position or policies of the U.S. Department of Justice.

**U.S. DEPARTMENT OF JUSTICE
LAW ENFORCEMENT ASSISTANCE ADMINISTRATION
NATIONAL CRIMINAL JUSTICE REFERENCE SERVICE
WASHINGTON, D.C. 20531**

Date filmed

6/2/76

Aerospace Report No.
ATR-76(7915)-1

EQUIPMENT SYSTEMS IMPROVEMENT PROGRAM

GUNSHOT RESIDUE DETECTION USING INORGANIC LUMINESCENCE

Prepared by

Peter F. Jones, Robert S. Nesbitt,
John E. Wessel, and Gerard M. Wolten
Chemistry and Physics Laboratory
Laboratory Operations

September 1975

Law Enforcement Development Group
THE AEROSPACE CORPORATION
El Segundo, California 90245

Prepared for

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

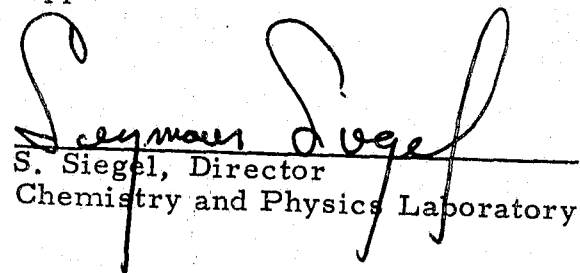
Contract No. J-LEAA-025-73

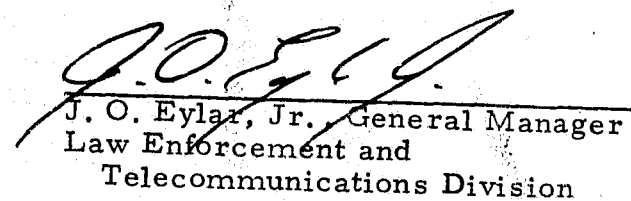
This project was supported by Contract Number J-LEAA-025-73 awarded by the National Institute of Law Enforcement and Criminal Justice, Law Enforcement Assistance Administration, U. S. Department of Justice, under the Omnibus Crime Control and Safe Streets Act of 1968, as amended. Points of view or opinions stated in this document are those of the authors and do not necessarily represent the official position or policies of the U. S. Department of Justice.

EQUIPMENT SYSTEMS IMPROVEMENT PROGRAM

GUNSHOT RESIDUE DETECTION USING INORGANIC LUMINESCENCE

Approved


S. Siegel, Director
Chemistry and Physics Laboratory


J. O. Eylar, Jr., General Manager
Law Enforcement and
Telecommunications Division

ABSTRACT

Over 170 gunshot residue handsamples were analyzed, using the inorganic luminescence detection method, in order to assess the potential for a gunshot residue test based on elemental analysis. Data were obtained for both indoor and outdoor firings using a variety of commercial brands of ammunition. The tape adhesive sample collection method, previously developed for detection by the particle analysis method based on the scanning electron microscope, was found very effective in collecting gunshot residue for analysis by inorganic luminescence, as well as minimizing sensitivity to background interference. Analyses of 45 handblanks gave results for antimony consistently lower than those based on neutron activation analysis that have been reported by others. However, amounts of antimony found in residue specimens using the inorganic luminescence method and obtained from firings of the .22, .32, and .380 caliber, and 9 mm semiautomatic pistols and .22, .32, and .38 caliber revolvers studied were comparable to those reported in other studies. The data suggest that lead is slightly more useful than barium as an indicator of the presence of gunshot residue. Some preliminary tests were made to measure persistence, and transfer of residue was studied. Lead declined to background levels when collection of residue was delayed for one hour after firing and participants were allowed unrestricted activity. Although antimony levels declined significantly, they tended to remain above background levels for up to four hours. Transfer of residue from the non-firing hand was frequently apparent when delays in collection of one, two, and

three hours were involved. Transfer of residue from hands to pockets was readily apparent from the antimony content of samples taken from the pocket. It was also observed, as reported by others, that although residue could be removed by wiping hands on clothing or with towels, detectable traces of antimony remained.

A simple, inexpensive, commercial filter fluorimeter was modified for antimony detection. Preliminary tests indicate that the instrument will perform well for the analysis of antimony. This conclusion is based on the antimony levels found in the limited persistence data obtained in this study, but can be modified as a result of more complete persistence data that will be obtained as part of further studies of the particle analysis method.

CONTENTS

ABSTRACT	v
ACKNOWLEDGEMENT	xi
SUMMARY	xiii
I. INTRODUCTION	1
II. EXPERIMENTAL PROCEDURE	5
III. RESULTS	15
A. .38 Special, Smith and Wesson	15
B. 9 mm Semiautomatic, Browning	15
C. .380 Semiautomatic, Browning	19
D. .32 Caliber Revolver, Unknown Make	19
E. .32 Caliber Semiautomatic, Llama	19
F. .22 Caliber Rifle, Harrington and Richardson	20
G. Handblanks	23
H. Persistence	25
I. Filter Fluorimeter	30
IV. DISCUSSION	35
A. Handsamples Collected Immediately After Firing	36
B. Handblanks	39
C. Residue on Clothing	40
D. Persistence of Residue on Hands	42
E. Adhesive Residue Collection	43
F. Filter Fluorimeter	44

CONTENTS (Continued)

V. CONCLUSION	47
NOTES	49
APPENDIX. HANDSAMPLE DATA	51
GLOSSARY	63

ILLUSTRATIONS

1. Tape Adhesive Collection Method	6
2. Photoluminescence Apparatus	8
3. Representative Low-Temperature Luminescence Excitation Spectra for Lead and Antimony Analyses of Handsamples	11
4. Modified Commercial Filter Fluorimeter	13
5. Lead Found in Handsamples from Indoor and Outdoor One-Round Firings of a .38 Special Smith and Wesson Revolver	17
6. Antimony Found in Handsamples from Indoor and Outdoor One-Round Firings of a .38 Special Smith and Wesson Revolver	18
7. Lead Found in Handsamples from One-Round Firings of a .32 Llama Semiautomatic Pistol	21
8. Antimony Found in Handsamples from One-Round Firings of a .32 Llama Semiautomatic Pistol	22
9. Lead Found in Handblank Samples Taken with Tape Adhesive Collection Method	24
10. Persistence with Time of Antimony on Firing Hand Following One-Round Firing of a .32 Llama Semiautomatic Pistol	31

TABLES

1. Summary of Data for Handsamples Collected Immediately After Firing and for Handblanks	16
2. Removal of Gunshot Residue by Placing Hands in Pockets	26
3. Effect of Wiping Hands on Clothing on Persistence of Gunshot Residue	28
4. Average of Lead and Antimony Found on Hands at Various Time Intervals Following Firing of a .32 Llama Semiautomatic Pistol	29
5. Effect of Time on Persistence of Gunshot Residue Studied with a Variety of Guns	32
A1. .38 Special Smith and Wesson Revolver	53
A2. 9 mm Browning Hi-Power Semiautomatic Pistol	54
A3. .380 Browning Semiautomatic Pistol	55
A4. .32 Revolver	56
A5. .32 Llama Semiautomatic Pistol	57
A6. .22 Harrington and Richardson Revolver	58
A7. .22 Hi-Standard Semiautomatic Pistol	59
A8. Handblanks	60
A9. Effect of Time on Persistence of Gunshot Residue	61

ACKNOWLEDGMENT

The support and encouragement of the National Institute of Law Enforcement and Criminal Justice is acknowledged, with special thanks to Joseph Kochanski, George Shollenberger, and John Sullivan.

SUMMARY

The typical criminalistics laboratory has great need of an inexpensive, simple test for the presence of gunshot residue on the hands of suspects. A previous report¹ described a technique for gunshot residue analysis based on detection of antimony and lead by photoluminescence, which offered advantages over existing elemental detection procedures in terms of cost and ease of analysis. The objectives of this study were to establish convenient operational procedures for elemental analyses and to acquire a data base for firings of representative handguns and for handblanks. In addition, studies of the persistence of residue deposits following firing and of transfer of residue from firing to nonfiring hands and to clothing have been initiated.

Development of a highly convenient and efficient residue collection procedure has been of major importance to test procedures. The tape adhesive collection procedure was initially developed for the highly promising technique of particle detection by scanning electron microscopy. In this work, the tape adhesive collection procedure has been adapted to elemental analysis and found to offer significant advantages over previous methods. It reduces response to background contamination without sacrifice of sensitivity to antimony and lead components of residue. In addition, the tape adhesive is readily available, collection is extremely simple, and sample work-up is easy and rapid. The residue is collected by repeatedly pressing a disc coated with adhesive against the hand, covering the areas of residue deposit. In order to prepare a sample for analysis, several drops of dilute

hydrochloric acid are placed on the sample disc for about three minutes, and then the solution is transferred to a quartz sample tube in which the measurements are made directly.

Extensive data were obtained for handsamples taken following firing of a range of handguns spanning types commonly encountered in criminalistic work. In particular, .22, .32, and .38 caliber revolvers and .22, .32, and .380 caliber, and 9 mm semiautomatic pistols were used. The measured post-firing quantities of antimony were in accord with those reported in previous studies that relied on neutron activation analysis. In addition, the data represents a significant addition to the information about the lead content of residue, which was not available from neutron activation analysis. Lead measurements varied over the range of 0.1 to 9.0 micrograms for firings, and over the range of 0.08 to 1.3 micrograms for handblanks. In contrast, antimony in firing samples varied from less than 0.01 to 2.2 micrograms, whereas it was normally undetectable (less than 0.01 micrograms) in handblanks. Only one handblank out of 45 had more than 0.01 micrograms of antimony, and it was taken from a machinist. Of all firing handsamples studied, 89 percent contained more than 0.01 micrograms of antimony. These results suggest that measurement of antimony by inorganic luminescence should be an effective screening test for the presence of gunshot residue. The data indicate that considerably less antimony is detected in handblanks using inorganic luminescence and the tape lift than was found earlier for a similar class of subjects using neutron activation analysis and a paraffin lift.

Limited studies were also made of persistence and transfer of residue. The amount of antimony on the firing hand decreased by an order of magnitude in the first hour and then more slowly. When a .32 Llama semiautomatic pistol was used, antimony generally could be detected on the firing hand four hours after the pistol was fired. Lead, however, was not detectable above the normal handblank value after one hour. Transfer of antimony to the nonfiring hand was also observed when sample collection was delayed.

The effect of the shooter's activity on the persistence of residue was tested by having subjects repeatedly place their hands inside their pants pockets after firing and before the collection of residue. Also, tests were made after subjects wiped their hands on clothing following firing. In both cases, lead was reduced to handblank levels, whereas antimony remained above handblank quantities. Samples were also taken directly from subjects' pants pockets after they had placed their firing hands inside their pockets three times. In each case, more antimony was detected than was measured in the highest pocketblank. Sampling of pants pockets may represent a useful new procedure because retention of residue in the pocket can be anticipated to be more prolonged than on hands.

Overall comparison of results with those reported in prior studies by neutron activation analysis supports the conclusion that antimony is the most valuable elemental indicator for the presence of residue. Both lead and barium are of limited value because of their higher abundance in handblanks. The new data suggest that lead is slightly more useful than barium.

A limited search was made of potential luminescence methods for detecting barium, but no promising approaches have been identified. Room-temperature methods of analysis for antimony and lead have also been sought, but the approaches pursued for antimony detection at room temperature lacked the required sensitivity, and the lead procedures were too sensitive to interference from background contamination. Since the existing low-temperature method is reliable and extremely easy to perform, further exploration of new methods has been deferred.

Preliminary testing of a commercial filter fluorimeter modified for antimony detection indicates that a simple, single-wavelength instrument can measure the antimony content of residue. Although the simple procedure is less specific for antimony than the measurement of the entire excitation spectrum, the sensitivity is definitely adequate. Therefore, there is excellent promise for a simple screening instrument, costing on the order of \$1000, provided spectral interference from typical handblank impurities is not encountered frequently at the wavelengths selected.

CHAPTER I. INTRODUCTION

This report summarizes results of a series of gunshot residue analyses that was conducted to evaluate the inorganic luminescence method for detection of antimony (Sb) and lead (Pb) in gunshot residue samples taken from the hands of suspects. Handblank and firing data have been obtained that will be useful for assessment of inorganic luminescence as a screening test for firearms cases. Methodology improvements are described that have been achieved since the initial report was issued.¹ This report, second in a series, is directed toward work with a variety of handguns, including a limited persistence study.

A new collection method for gunshot residue using a layer of tape adhesive has been developed as a replacement for the wash procedure used in the original work. In the studies described in this report, handsamples were taken immediately after one-round firings of various common caliber handguns, particularly .22, .32, and .38 calibers. These samples, as well as handsamples taken from persons who had not fired a gun (termed handblanks), were analyzed for Pb and Sb content. The persistence of gunshot residue on the back of the hands was studied as a function of activity and time delay of sample collection following one-round firings from a .32 caliber semiautomatic pistol. Pockets and shirt sleeves of clothing were also examined for evidence of gunshot residue, with positive results. This report is a substantial addition to the published data on Pb in residue

and is the first extensive quantitative survey based on the tape adhesive collection method.

Current detection procedures are based on elemental analyses. Neutron activation analysis is used to determine the presence of Sb and barium (Ba), whereas flameless atomic absorption spectrometry, which is currently being explored by several laboratories, is capable of detecting Sb, Ba, and Pb. The principal limitation of these procedures is the presence of these elements in environmental contaminants. Samples taken from the hands of persons who have not fired guns often show appreciable quantities of Ba and Pb. Therefore, in practice, a threshold limit is set that, if exceeded, is taken to signal a positive test for gunshot residue. Unfortunately, if this threshold is set high enough to avoid a significant number of false positive decisions (e.g., less than 5 percent false positives), then a large fraction of the decisions is falsely negative for known firing cases, even for samples collected immediately after firing.² This problem can now be overcome by use of the highly reliable detection method based on scanning electron microscopy, which was described in a recent report.³ It was shown that the scanning electron microscope with x-ray elemental analysis should provide highly specific identification of residue particles. The most recent data suggest that it will be possible to find and identify characteristic particles conclusively many hours after firing, even for small-caliber, clean guns, using this new detection method. This type of analysis requires expensive equipment and, as a result, many smaller crime laboratories would have to send their samples elsewhere. Thus, a simple

and less expensive elemental method of detection would be useful for local laboratory use as a screening test. For this application, the threshold limits set for positive results can be lowered substantially because positive samples would be submitted for confirmatory analysis using the scanning electron microscope. When the lower threshold is used, the occurrence of false negatives can be reduced to an acceptable frequency. For example, if the threshold is set to give no more than 5 percent false negatives for immediately collected samples of .38 caliber Special revolver firings, then, by extrapolation of published data, about 10 percent false positives would result for normal handblanks from subjects with low background exposure to Sb and Ba.

Some preliminary effort has been devoted to development of an inexpensive filter fluorimeter instrument for inorganic luminescence analysis. This instrument is intended as a screening detector for the small, local laboratory. Ideally, the cost would be within the budget limits of most laboratories (about \$1000), and little specialized training would be required for reliable operation.

CHAPTER II. EXPERIMENTAL PROCEDURE

After the firing of a gun, the backs of both hands (including the backs of the thumb and fingers and the web area between thumb and fore-finger) were sampled separately with a 2.5-cm-diameter disc that was coated on one side with Scotch No. 465 adhesive transfer tape. This collection procedure was chosen because the same discs could be inserted into the scanning electron microscope for analysis. Figure 1a shows the use of the adhesive-coated, disc-shaped sample block for collection of residue from the back of the hand.

For the photoluminescence analysis of Pb and Sb, 0.5 ml of 7 molar hydrochloric acid (HCl) was pipetted onto the tape surface and allowed to soak and react for 3 min. For periods longer than 3 min, the HCl began to react vigorously with the aluminum discs that were used to support the adhesive. After the HCl reaction, a Pasteur pipet was used to remove the acid from the tape, as shown in Figure 1b. Then the acid sample was placed in a Suprasil quartz sample tube of precision bore, 4-mm inside diameter and 6-mm outside diameter.* After the HCl-residue sample was pipetted into the specimen tube, the latter was immersed into liquid nitrogen

*Commercial grade quartz fluoresces strongly under ultraviolet excitation and therefore is unsuitable for use in this analysis. The quartz must have high transmission in the ultraviolet spectral region because the Sb excitation spectrum peaks at 240 nm. All optical components, such as filters and optical Dewars, must have low fluorescence.

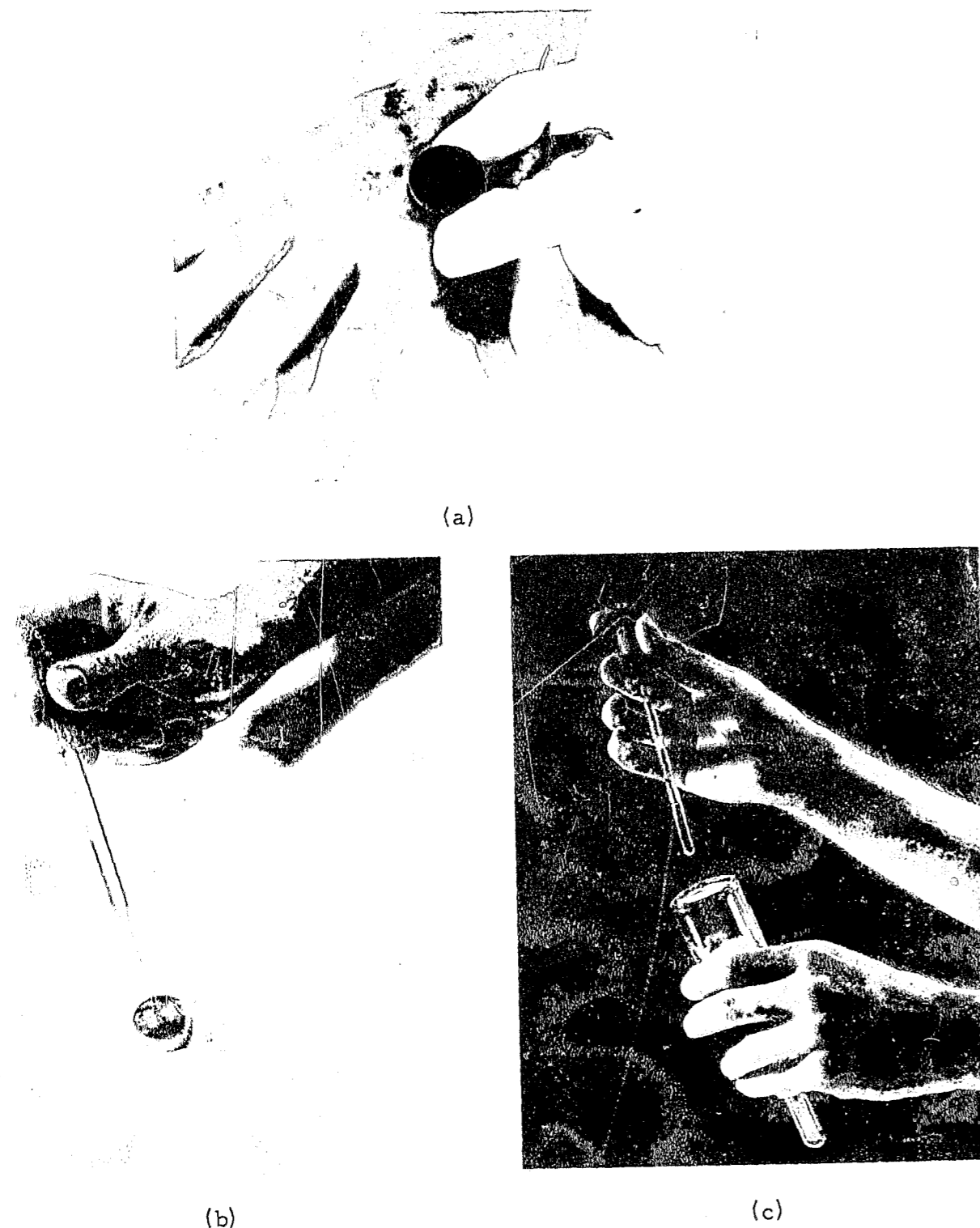


Fig. 1. Tape Adhesive Collection Method. (a) Disc-shaped sampling block, coated on one side with adhesive transfer tape, is applied to skin of back of hand. (b) Pasteur pipet is used to remove acid from tape sample after reacting for 3 min with gunshot residue. (c) The 0.5-ml HCl-gunshot residue sample has been removed from tape and pipetted into quartz sample tube, and is ready for immersion into liquid nitrogen in the Dewar flask, which is being held below.

in a quartz optical Dewar, as shown in Figure 1c, and was analyzed for Pb and Sb content. The quartz optical Dewar used is commercially available from manufacturers of fluorescence instrumentation, such as American Instrument Co., Inc. (Part No. B28-62140). The Dewar also can be specially fabricated by a local glass blower; it is of critical importance that the tip be designed for low fluorescence. Our Dewars were fabricated by Cal Glass Research, Inc., Costa Mesa, California. Following the Pb and Sb analysis at 77°K (boiling point of liquid nitrogen), the quartz sample tube must be rapidly heated to prevent breaking and loss of the tube as a result of the difference in coefficients of expansion between the frozen acid and the quartz.

Apart from the sample compartment, the photoluminescence instrumentation, shown in Figure 2, consisted of an excitation system and emission monitor (light detection) system. The excitation source used in most of this work was an Osram XBO 450 W/4 Suprasil quartz xenon direct-current arc lamp, chosen for its brightness and because its spectral output does not vary rapidly with wavelength. The smooth distribution of light output versus wavelength, characteristic of the xenon lamp, was required because the data were recorded in the form of excitation spectra, in which the luminescence intensity at a fixed wavelength is measured as the excitation wavelength is varied. The excitation light was directed through a continuous scanning excitation monochromator (Jarrell Ash Model 82-440 double monochromator), which selects the wavelength of light striking the sample. Focusing lenses were placed between the light source and the entrance slit of the excitation

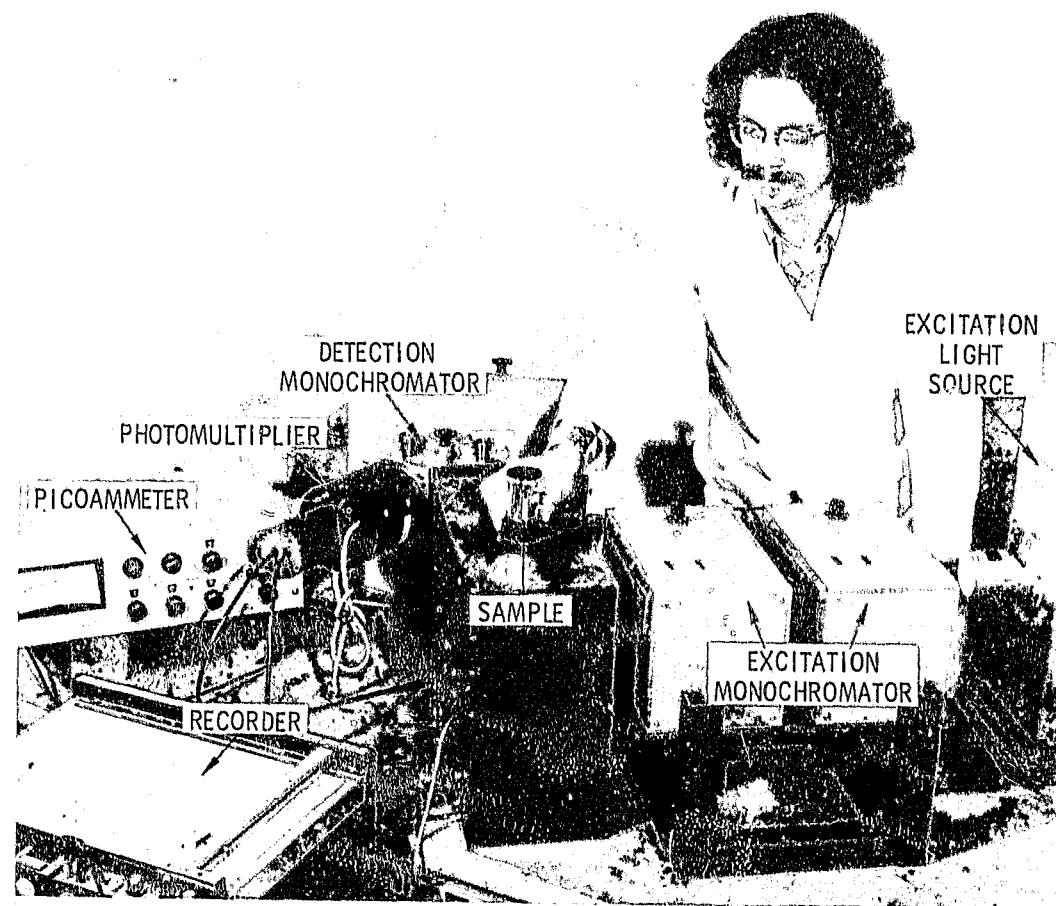


Fig. 2. Photoluminescence Apparatus

monochromator, and between the exit slit of the excitation monochromator and the sample. The excitation light induces luminescence from the sample, which was focused onto the slits of an emission monochromator (McPherson model 218, 0.3 meter) that was preset to pass the wavelength characteristic of the luminescence of the constituent to be measured. The luminescence passing through the emission monochromator was detected with the red-sensitive RCA model C 31034 photomultiplier, which has a flat spectral response from 400 to 700 nm. The photomultiplier signal was amplified with a Keithley 417 picoammeter, and the spectra recorded with a Houston Omnigraphic model 2000 X-Y recorder. The instrumentation is of modular arrangement, and appropriate optical filters could readily be used to replace the emission monochromator, thereby simplifying the system. The modular arrangement also allows for any comparable combination of light sources, monochromators, amplifiers, photomultipliers, and recorders of different manufacture and model number to be employed in this analysis.

For Sb analysis, the emission monochromator was set at 660 nm, although the Sb emission peaks at about 600 to 620 nm. The selection of 660 nm was made to minimize background interference. An optical cut-off filter of low fluorescence, Schott FG-10, was placed between the sample compartment and the emission monochromator in order to exclude any scattered excitation light from reaching the monochromator. For Pb analysis, the emission monochromator was set at the emission maximum, 385 nm, and the use of filters was not required. With the emission monochromator set at 660 nm for Sb and at 385 nm for Pb, and with continuous

variation of the excitation wavelength, maxima were observed at 240 nm and 300 nm for Sb and at 272 nm for Pb. These excitation spectra are analogous to the absorption spectra after correction for the variation with wavelength of the intensity of the excitation source. Figure 3 exhibits representative data (excitation spectra) for Pb and Sb analyses of hand-samples taken from the firing hand after a one-round, .38 Special revolver firing; after a one-round, .22 revolver firing; and of a handblank. For quantitative analysis, calibration plots are made from Pb and Sb standard solutions in HCl. The acid-residue samples are analyzed shortly after preparation because the Pb and Sb complexes begin to lose luminescence intensity after a period of time. A. C. S. reagent grade HCl from Baker and Adamson (Allied Chemical) was used successfully without background interference or luminescence quenching. The detection limits of Sb and Pb in the absence of organic contaminants from the hand are 0.5 and 0.1 ng, respectively. In the presence of typical organic hand contaminants, the detection limit for both Sb and Pb is 10 ng.

Barium does not form a luminescent molecular complex with HCl. No satisfactory fluorometric analysis method for Ba that could be applied for gunshot residue analysis has yet been developed. Additional details concerning the analysis method are contained in the original report.¹

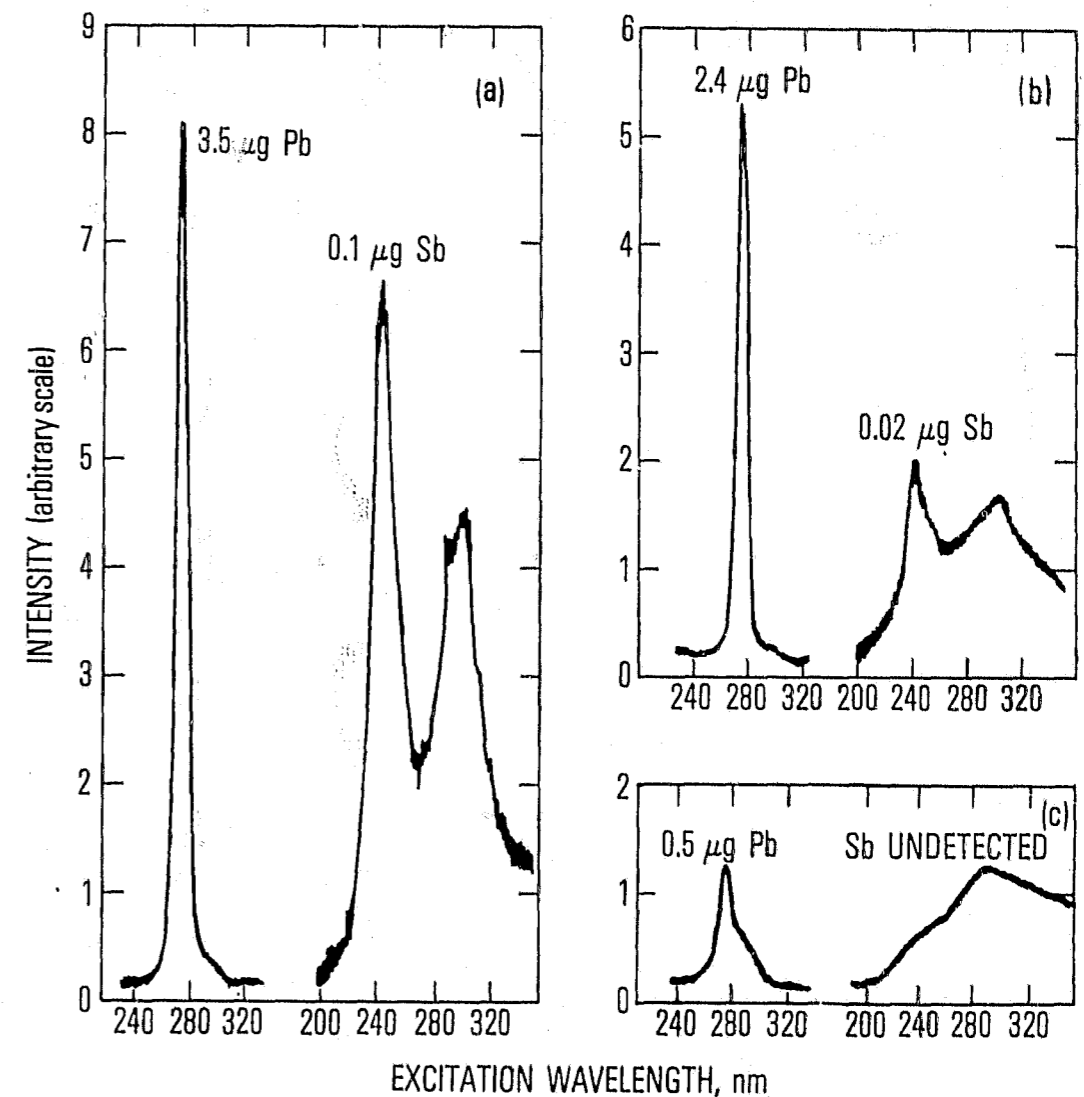


Fig. 3. Representative Low-Temperature Luminescence Excitation Spectra for Lead and Antimony Analyses of Handsamples: (a) After a one-round .38 Special revolver firing, (b) After a one-round .22 revolver firing, and (c) Of a handblank. Sb emission was monitored at 660 nm while the excitation was scanned, and Pb emission was monitored at 385 nm during the excitation scan.

Some analyses were also made using a Baird Atomic Fluorimet filter fluorimeter (Figure 4) that was modified to permit antimony analysis. The 254-nm mercury resonance line was used for excitation, and an interference filter centered at 250 nm was inserted between the mercury lamp and the sample to prevent all excitation except the 254-nm mercury line from reaching the sample. A holder block was machined to position the liquid nitrogen Dewar rigidly. The block was provided with slits that confined excitation and detection to the desired sample area. An interference filter centered at 622 nm with 27-nm full width at half height was used to isolate Sb luminescence. The Hamamatsu R446 photomultiplier supplied with the fluorimeter was used without modification. Sample preparation was the same as for the other work involving the spectrofluorimeter. Data were reduced by subtracting the signals for reagent blanks from those for handsamples.

Most guns used in the study were new. They included a .22 caliber Harrington and Richardson model 929 revolver (2-in. barrel), a .22 caliber Hi-Standard model M-101 semiautomatic pistol, a .32 caliber revolver of unknown make and very poor condition, a .32 caliber Llama semiautomatic pistol, a .380 Browning semiautomatic pistol, a 9 mm Browning Hi-Power semiautomatic pistol, and a .38 Special Smith and Wesson revolver (2-in. barrel). All of the 168 test firings were single-round firings using commercial brand ammunition. Analysis of primers from .22 long rifle ammunition by means of the photoluminescence method (which only detects Pb and Sb) indicated that the Federal primer contained Pb and Sb, whereas Western, Browning, C. C. I., and Remington contained Pb but not Sb.

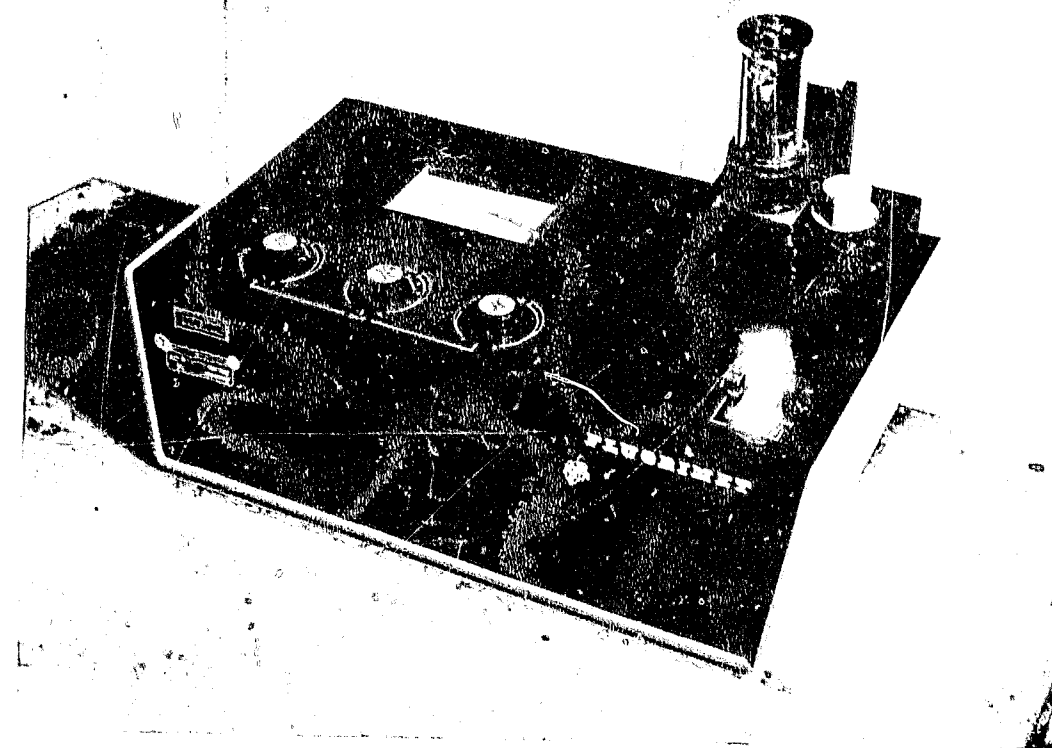


Fig. 4. Modified Commercial Filter Fluorimeter

CHAPTER III. RESULTS

In this Chapter, the amounts of lead and antimony found are summarized, weapon by weapon, in Sections A through D. In addition to the text, there are tables and figures. Section E contains the data for hand-blanks, and Section G describes the results of a limited persistence study. The final section, H, describes results obtained with a low-cost filter fluorimeter instead of the more elaborate instrument described in Chapter II.

A. .38 Special, Smith and Wesson

Average amounts of Pb and Sb found in firing handsamples taken after one-round firings of a .38 Special Smith and Wesson revolver are listed in Table 1, and original data are presented in the Appendix. For 31 outdoor firing samples, the Pb and Sb averages were 0.6 and 0.03 μg , respectively. For 16 indoor firings, the Pb and Sb averages were 4 and 0.13 μg , respectively. As shown by the Pb and Sb averages and by the graphical displays in Figures 5 and 6, the indoor firing samples produced greater amounts of Pb and Sb than did the outdoor firing samples. None of the 16 indoor firing samples gave Pb and Sb amounts less than the highest Pb and Sb amounts seen in our handblank analyses.

B. 9 mm Semiautomatic, Browning

Data on Pb and Sb for outdoor and indoor firing handsamples taken following one-round firings of a 9 mm Browning Hi-Power semiautomatic pistol are listed in Table 1. The nine outdoor samples showed average

Table 1. Summary of Data for Handsamples Collected Immediately After Firing^a and for Handblanks

Handgun	Outdoor			Indoor		
	Average		Number Samples	Average		Number Samples
	Pb (μg)	Sb (μg)		Pb (μg)	Sb (μg)	
Revolvers						
.38 Special	0.6	0.03	31	4.0	0.13	16
.32	1.3	0.05	3	3.0	0.07	2
.22	1.0	0.03	13	1.4	0.05	6
Semiautomatic Pistols						
9 mm	0.5	0.02	9	2.2	0.1	2
.380	2.2	0.7	6	5.0	0.5	5
.32				2.3	0.8	20
.22	0.5	0.03	2	1.9	0.07	9
Handblanks	0.4	<0.01	45			

^aData appear in Tables 1 through 8 of the Appendix.

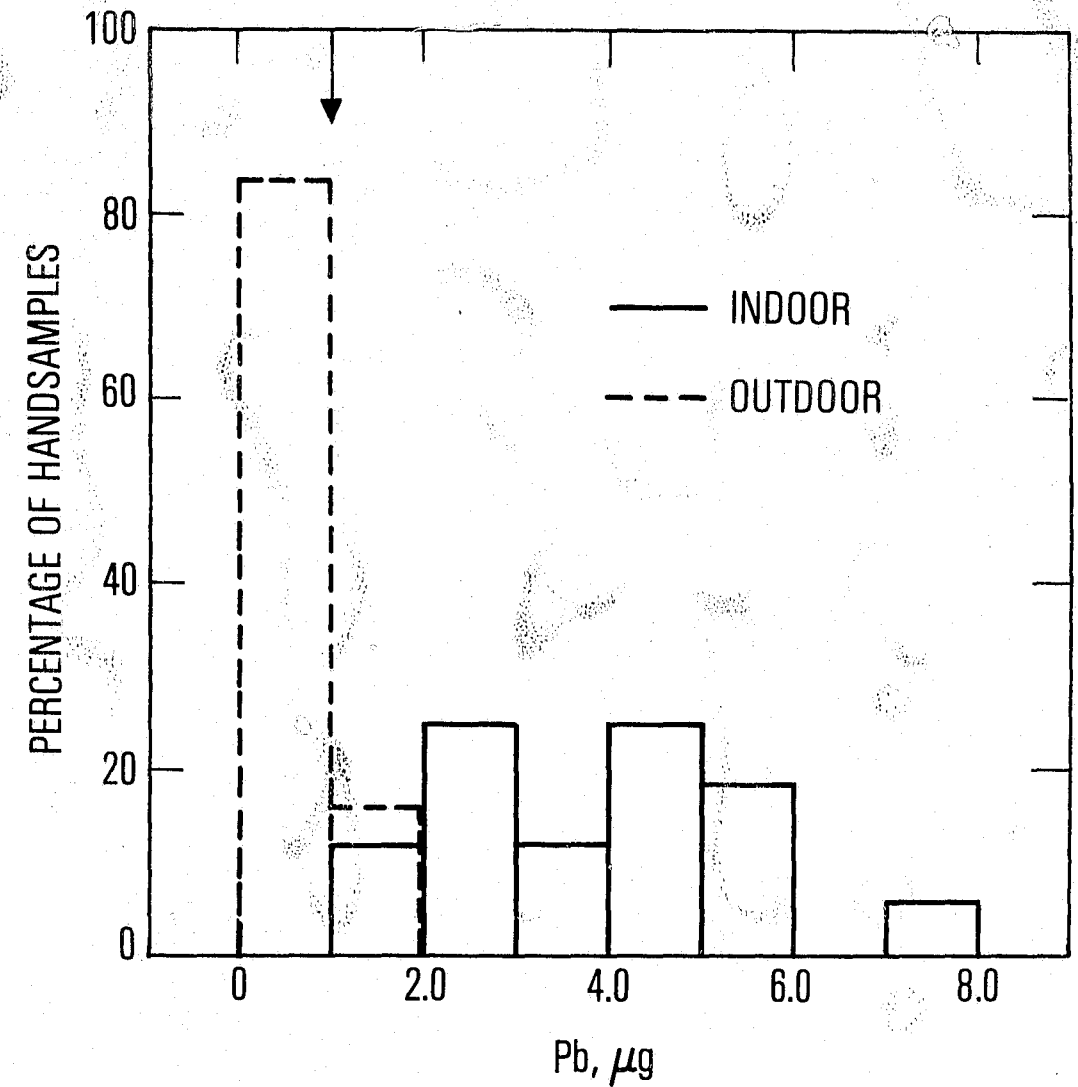


Fig. 5. Lead Found in Handsamples from Indoor and Outdoor One-Round Firings of a .38 Special Smith and Wesson Revolver. Samples were collected with tape. 44 of the 45 handblanks show Pb levels below 1.0 μg , as indicated by arrow. The vertical coordinate in the figure refers to the percentage of analyses results falling within the range shown on the horizontal coordinate.

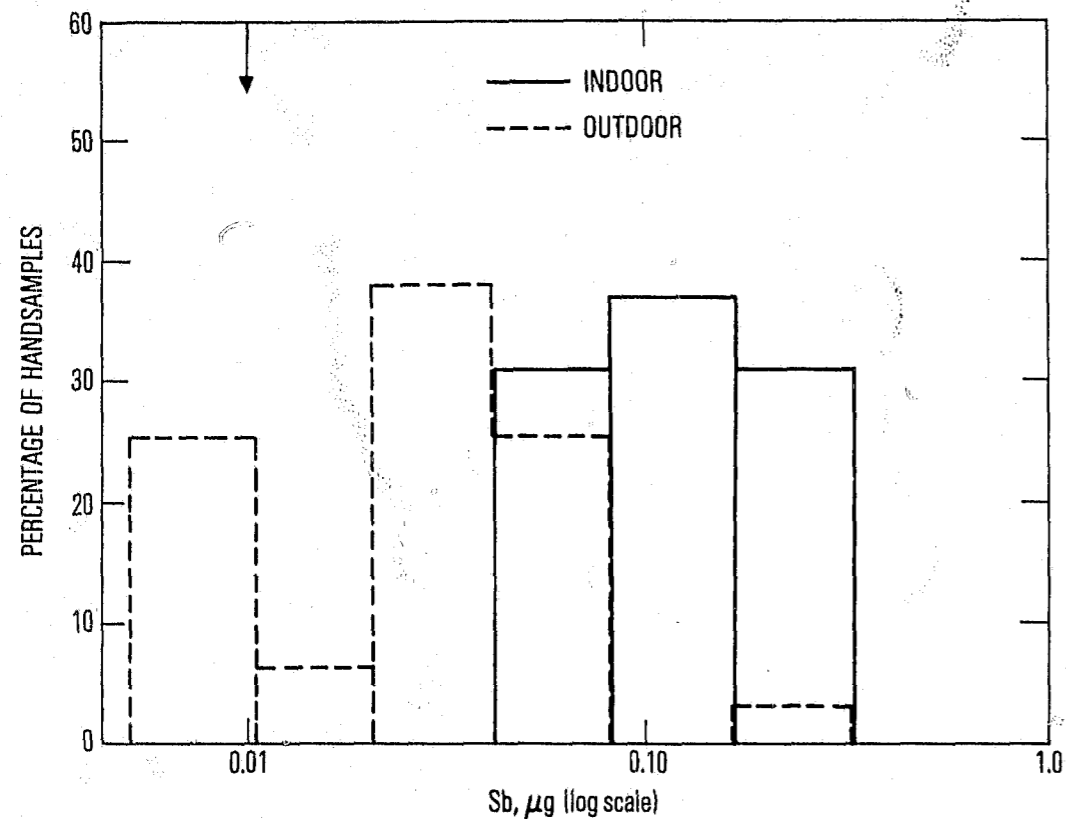


Fig. 6. Antimony Found in Handsamples from Indoor and Outdoor One-Round Firings of a .38 Special Smith and Wesson Revolver. Samples were collected with tape. 44 of the 45 handblanks show Sb levels below 0.01 µg, as indicated by arrow. The logarithmic scale of the abscissa (amounts of Sb) shows the separation of indoor and outdoor firings more clearly than a linear scale would.

Pb and Sb amounts of 0.5 and 0.02 µg, respectively. These two averages do little to distinguish these handsamples from handblanks. Only two indoor firing samples were obtained with the 9 mm semiautomatic pistol, and they both contained 2 µg Pb and 0.1 µg Sb. If they are typical of the average Pb and Sb amounts found on the back of the firing hand after firing the gun indoors, then a disparity between the indoor and outdoor samples is again indicated.

C. .380 Semiautomatic, Browning (.380 ACP or 9 mm Short Ammunition)

Our previously published preliminary work centered on the analysis of handsamples taken immediately following two-round firings of a .380 caliber Browning semiautomatic pistol. This pistol leaves a large amount of gunshot residue on the hands after firing and is an uncommon handgun in crimes involving firearms.

In our early work on the photoluminescence analysis for Pb and Sb in gunshot residue, the backs of the hands were sampled with a water rinse. The 28 samples from the two-round firings at an outdoor pistol range gave Pb and Sb averages of 1.1 and 0.09 µg, respectively. An additional 11 residue samples have been collected from hands immediately following one-round firings using the tape method. The average amount of Pb for the six outdoor firings was 2.2 µg and for Sb was 0.7 µg. Five indoor firings yielded an average of 5 µg Pb and 0.5 µg Sb. These figures corroborate our earlier findings that this gun produces large amounts of residue.

D. .32 Caliber Revolver, Unknown Make

The Pb and Sb amounts found in handsamples taken immediately after one-round firings of a .32 caliber revolver are shown in Table 1 and the

Appendix. The make of this revolver was unknown, and because of the very poor operating condition of the gun, work with it was discontinued after limited use. Only three one-round firings were made outdoors and two firings indoors with this gun.

E. .32 Caliber Semiautomatic, Llama

The amounts of Pb and Sb found on the back of the firing hand immediately after firing one round from a .32 caliber Llama semiautomatic pistol are shown in Table 1. For 20 indoor firing handsamples, the Pb and Sb averages were 2.3 and 0.8 μg , respectively. Figures 7 and 8 give a graphical representation of the Pb and Sb amounts in tape collection samples. Handblank data from tape-collection samples are also included in the graphs in Figures 7 and 8. The semilogarithmic plot in Figure 8 emphasizes the marked difference between handblank amounts and shooting sample amounts. All of the handblanks gave less than 0.03 μg Sb, while all of the shooting handsamples exceeded this amount. One hundred percent of the Pb handblank values fell below 1.3 μg , while 85 percent of the firing samples using the .32 caliber semiautomatic pistol were above 1.3 μg Pb.

F. .22 Caliber Revolver, Harrington and Richardson

Table 1 shows Pb and Sb data for firing handsamples from a 2-in. - barrel Harrington and Richardson .22 caliber revolver. The brands of .22 long rifle ammunition are listed with the analysis data in the Appendix. Only the Federal brand cartridge primers were found to contain Sb. The obvious presence of Sb in gunshot residue samples collected after firing of antimony-free cartridges may be caused by contamination from the Federal ammunition previously fired from the same gun, or it may be from the Sb

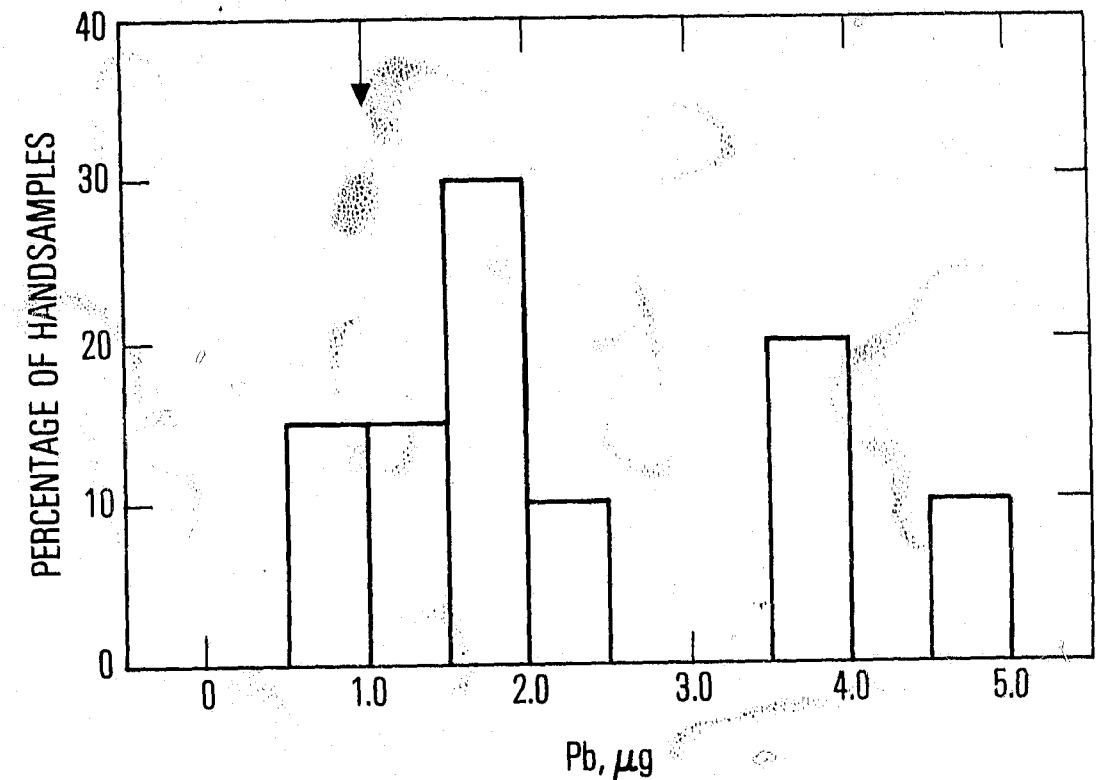


Fig. 7. Lead Found in Handsamples from Indoor One-Round Firings of a .32 Llama Semiautomatic Pistol. Samples were collected with tape. 44 of the 45 handblanks show Pb levels below 1.0 μg , as indicated by arrow.

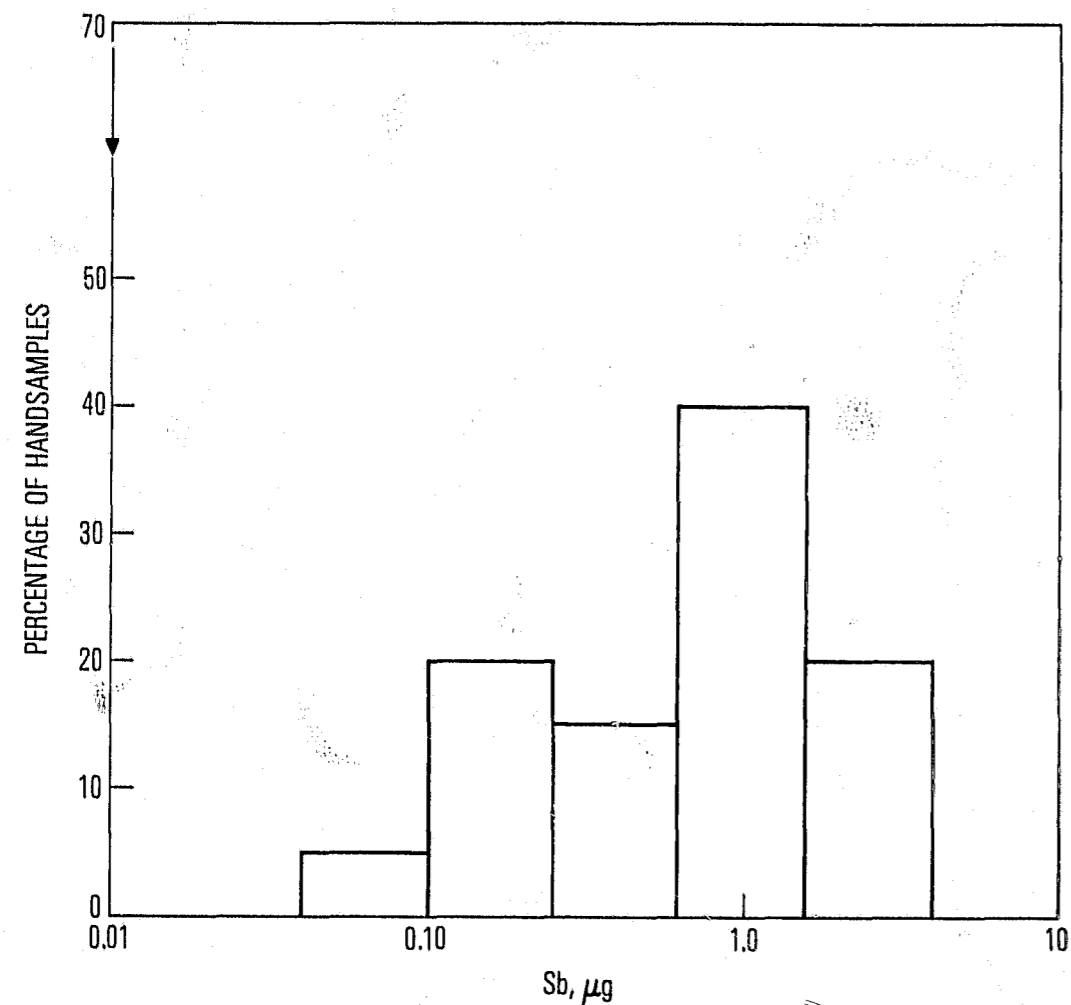


Fig. 8. Antimony Found in Handsamples from Indoor One-Round Firings of a .32 Llama Semiautomatic Pistol. Samples were collected with tape. 44 of the 45 handblanks show Sb levels below 0.01 µg, as indicated by arrow. The logarithmic scale for amounts of Sb emphasizes the separation of firing samples (handsamples) from the handblanks.

in the bullet. The average amount of Pb found in the 13 outdoor firing samples was 1.0 µg, and the Pb average in seven indoor firing samples was 1.4 µg. The average Sb amount found in the 13 outdoor samples, four of which were from Federal ammunition, was 0.03 µg. The Sb average for eight indoor samples, two of which were from Federal ammunition, was 0.05 µg.

Table 1 displays Pb and Sb amounts found in samples from firings of a .22 Hi-Standard semiautomatic pistol. Only two firings were made outdoors using this gun. The average Pb and Sb amounts for nine indoor firings were 1.9 and 0.07 µg, respectively.

G. Handblanks

In our previous work,¹ 20 handblank samples were taken by the water-wash collection method, and Pb and Sb analyses were made. No Sb was observed in any of the wash samples, all taken from laboratory workers. The Pb average for these samples was 0.2 µg, with the highest Pb value being 0.6 µg.

With the tape collection method, 45 handblanks were taken and analyzed for Pb and Sb content. Detectable Sb (amounts equal to and greater than 0.01 µg) was observed in just one tape-lift handblank, 0.03 µg, and the Pb average was 0.4 µg. Table 1 and the Appendix give a list of Pb and Sb amounts found in tape-lift samples taken from the backs of the hands of laboratory workers, painters, machinists, auto mechanics, maintenance men, and workers in other fields, while at work. Just one Pb value above 1.0 µg is seen. Figure 9 shows the distribution of the percentage of the

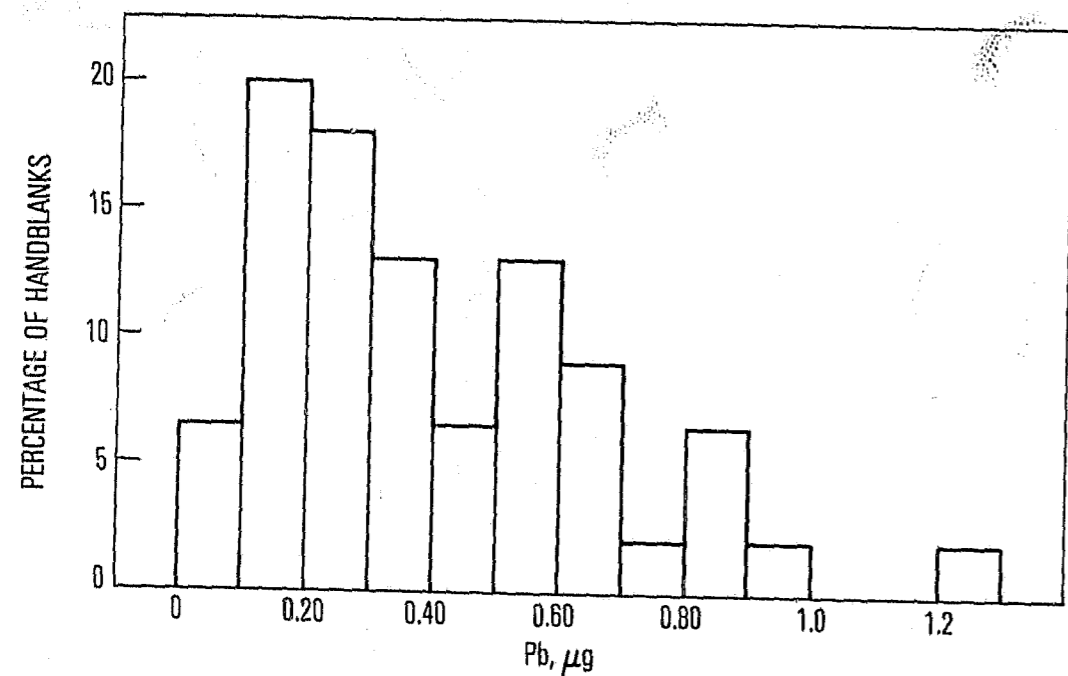


Fig. 9. Lead Found in Handblank Samples Taken with Tape Adhesive Collection Method.

number of handblank samples analyzed versus Pb amount for the tape-collection handblanks.

H. Persistence

A study was made of the effect of activity and elapsed time after shooting on the persistence of gunshot residue on the back of the hands. The major tests were carried out with one-round, one-hand, indoor firings of a .32 Llama semiautomatic pistol using Federal brand ammunition (cartridges with 71-grain metal case bullets). Aspects of this study were prompted by the work of Kilty.⁴

In the first persistence experiment, one round was fired with the .32 caliber pistol, followed by placement of the firing hand into a front pants pocket three times. The back of the firing hand was then sampled with tape, and the tape sample was analyzed for Pb and Sb; Table 2 lists the data. The pocket into which the firing hand was placed three times was also sampled, and Table 2 shows the Pb and Sb analysis data. Blank pocket samples, taken from pockets that had no previous contact with gunshot residue, were collected from the same persons whose pockets had been sampled during the persistence experiment. The inside of the lip of the pocket was the primary area that was sampled for gunshot residue. The Pb and Sb averages of the seven handsamples taken after placement into a pants pocket three times were 0.9 and 0.12 μg , respectively. The Pb average may not be of significance when compared to handblank data, but the Sb average is significantly above handblank values. One can compare these Pb and Sb handsample averages with the averages of Pb (2.3 μg) and

Table 2. Removal of Gunshot Residue by Placing Hands in Pockets

A. Handsamples of Firing Hands After Placement Into Pocket Three Times ^a			B. Samples Taken From Pockets After Firing Hand Was Placed Inside Pocket Three Times ^c			C. Samples Taken of Clean Pockets That Contain No Gunshot Residue		
Subject	Pb (µg)	Sb (µg)	Subject	Pb (µg)	Sb (µg)	Subject	Pb (µg)	Sb (µg)
1	1.4	0.16	1	1.1	0.09	1	0.13	- ^d
2	0.78	0.11	2	0.43	0.10	2	0.19	-
3	0.55	0.20	3	1.7	0.08	3	0.15	-
4	1.0	0.07	4	0.40	0.07	4	0.33	-
5	0.52	0.14	5	0.55	0.10	5	0.88	-
6	0.22	0.01	6	0.93	0.13	6	0.14	- ^d
7	1.7	0.18	7	0.47	0.12	7	0.17	- ^d
Average	0.9 ^b	0.12 ^b	Average	0.8	0.10	Average	0.28	-

^aFollowing the firing with one hand ~~in~~ one round from a .32 caliber Llama semiautomatic pistol, the firing hand was placed in the front pants pocket three times. Then the firing hand was sampled for gunshot residue with the tape adhesive collection method.

^bThe average amounts of Pb and Sb found immediately after firing were 2.3 and 0.8 µg, respectively.

^cFollowing the firing with one hand of one round from a .32 caliber Llama semiautomatic pistol, the firing hand was placed three times into the front pants pocket. Then the inside of the pocket was sampled for gunshot residue using the tape adhesive collection method. The main area of the pocket sampled was the inside of the lip.

^dEvidence suggesting trace amounts of Sb was seen, but the quantity was less than 0.01 µg.

Sb (0.78 µg) in handsamples taken immediately after firing the .32 caliber semiautomatic pistol.

Residue was also found in pockets. Data shown in Table 2B refer to the average quantity of Pb and Sb contained in samples taken from the inside lip of the pockets of subjects who fired. Following one-round firings of a .32 caliber semiautomatic pistol, subjects placed their firing hands into their pockets three times. Tape adhesive mounted on 1-in.-diam sampling discs was then pressed against the pockets to collect the residue. The average amount of Pb was 0.8 µg and that of Sb was 0.10 µg, whereas for residue-free pockets, Pb averaged 0.28 µg and Sb was less than 0.01 µg (data are tabulated in Table 2C).

One-round, one-hand, indoor firings with Federal ammunition were conducted with the .32 caliber Llama semiautomatic pistol, followed by vigorous wiping of the hands on clothing for about 15 sec, in order to effect the removal of the residue from the hands. The average amounts of Pb and Sb found on the firing handsamples after this activity were 0.6 and 0.04 µg, respectively. The data for the seven samples analyzed are listed in Table 3.

Tests of the effect of normal activity on persistence of residue were made for one-round, one-hand, indoor firings of the .32 caliber semiautomatic pistol, which were followed by unrestricted activity for specified periods of time (except that handwashing was forbidden). The backs of both hands were sampled with tape following 1-, 2-, 3-, and 4-hr time delays. Table 4 lists the amounts of Pb and Sb found on the handsamples after the different time periods. The largest declines in amounts

Table 3. Effect of Wiping Hands on Clothing on Persistence of Gunshot Residue^a

Subject	Pb (μg)	Sb (μg)
1	1.0	0.03
2	0.69	0.03
3	1.2	0.07
4	0.35	-
5	0.35	0.01
6	0.35	-
7	0.41	0.12
Average	0.6	0.04

^aFollowing the firing of one round from a .32 Llama semiautomatic pistol, the hands were wiped on the clothing vigorously for about 15 sec in an attempt to remove the gunshot residue. Then the firing hand was sampled by the tape adhesive collection method.

Table 4. Averages of Pb and Sb Found on Hands at Various Time Intervals Following Firing of a .32 Llama Semiautomatic Pistol^a

Time Period (hr)	Firing Hand		Nonfiring Hand	
	Pb (μg)	Sb (μg)	Pb (μg)	Sb (μg)
0	2.3 ^b	0.8 ^b	0.5	-
1	0.6	0.08	0.5	0.01
2	0.8	0.06	0.5	0.01
3	0.8	0.04	0.6	0.01
4	0.6	0.03	0.4	-

^aThe Pb and Sb averages come from data listed in Table A9.

^bThe Pb and Sb averages come from Table A5.

of Pb and Sb were seen between the immediate (0-hr) and 1-hr samplings. For the 1-hr time interval and longer, the Pb levels were indistinguishable from background levels. The average Sb amounts on the tape samples taken from the backs of the firing hands dropped by an order of magnitude between 0-hr and 1-hr samplings. As the time interval between firing and sampling lengthened, the average Sb amount from firing handsamples decreased, but at a slower rate after the first hour. Figure 10 shows a plot of average Sb amounts versus time for the delay of sampling following the firing. The nonfiring handsamples gave undetectable levels at the 0- and 4-hr intervals, while for each of the 1-, 2-, and 3-hr time intervals after firings, two of the five samples taken contained detectable Sb. Table 4 displays these averages for Pb and Sb.

Data on Pb and Sb from limited persistence tests using several different guns and makes of ammunition are shown in Table 5. During the time interval between the firing (one round with one hand, indoors) and sample collection, unrestricted activity was allowed except that hands were not washed.

I. Filter Fluorimeter

Some preliminary effort has been devoted to development of an inexpensive filter fluorimeter instrument for inorganic luminescence analysis. This instrument is intended as a screening detector for the small, local laboratory.

Six single-round firing specimens obtained with indoor firings of a .38 caliber Special revolver were analyzed by both the filter fluorimeter

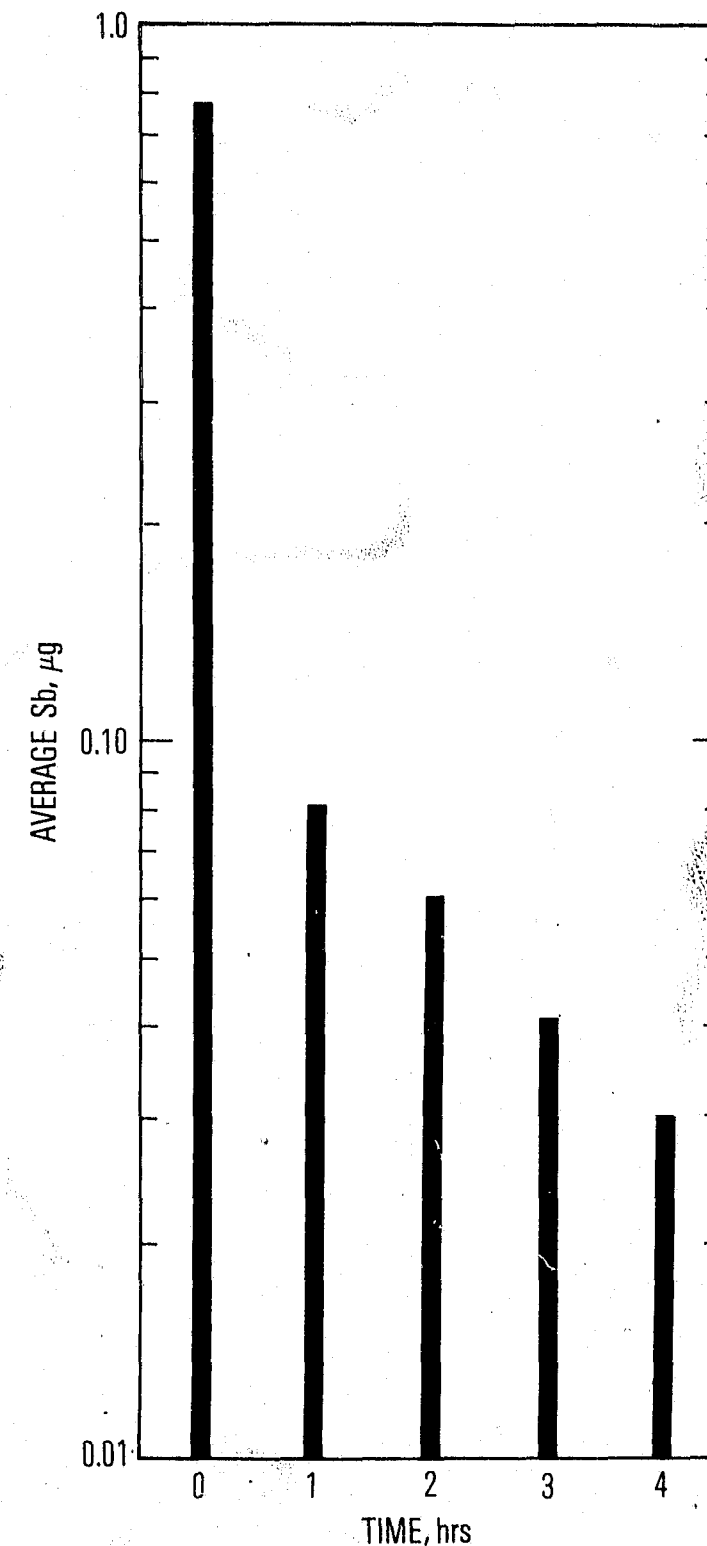


Fig. 10. Persistence with Time of Sb on Firing Hand Following One-Round Firing of a .32 Llama Semiautomatic Pistol. Samples were collected with tape.

Table 5. Effect of Time on Persistence of Gunshot Residue Studied with a Variety of Guns

Time Period (hr)	Handgun Used	Ammo Brand	Pb (μg)	Sb (μg)
1	.38 Special Smith and Wesson Revolver	Remington	1.1	0.05
1	.38 Special Smith and Wesson Revolver	Remington	1.0	0.03
1	.32 Revolver	Western	0.8	0.02
1	.32 Revolver	Western	0.9	0.02
1	.22 Hi-Standard Semiautomatic	Remington	2.0	0.08
2	.22 Hi-Standard Semiautomatic	Western Super X	1.1	0.01
2	.22 Hi-Standard Semiautomatic	Federal	1.5	0.03
2	.22 Smith and Wesson Revolver	Browning	0.6	-
2	.38 Special Smith and Wesson Revolver	Norma	3.0	0.13
2	.38 Special Smith and Wesson Revolver	Norma	2.9	0.09

and the recording spectrofluorimeter that has been used for past work. The same amounts of Sb were observed in each case. Six handblanks were also examined. The handblanks showed no evidence of Sb in the spectrofluorimetric analysis, although emission signals were observed with the filter fluorimeter, the highest being equivalent to the 0.03- μg standard. Because the filter fluorimeter only looks at light signals in a particular wavelength region (e.g., about 622 nm), low levels of Sb are not distinguished from background emission at 622 nm. With the spectrofluorimeter, the advantage of spectral shape makes Sb analysis more sensitive and definitive than the filter system.

CHAPTER IV. DISCUSSION

In previous studies, it was established that gunshot residue contains a heterogeneous mixture of particles that contain Pb, Sb, and Ba. In a prior paper that presented results obtained by the scanning electron microscope analysis of individual particles,³ it was concluded that some individual particles contain primarily one of these elements, whereas others contain mixtures. Furthermore, the size of the particles was observed to vary from less than 1 μm to greater than 100 μm . Therefore, any process that segregates particles on the basis of physical properties such as mass or density, for example, could be expected to influence the average quantities measured in an analysis of the residue that is finally collected. Factors involved in residue transport, such as wind, residue retention, or skin condition, as well as collection method, can potentially influence the average composition of the residue that is subjected to analysis. In fact, other workers have concluded that the wide variation in amounts and compositions of residues observed for successive similar firing tests can be explained on the basis that most of the mass of the elements detected is contained in a few large particles.⁵ It is then hypothesized that a large variation occurs in the number and composition of these larger particles recovered from firing to firing. Similar conclusions are supported by the data obtained in this study.

A. Handsamples Collected Immediately After Firing

In the .38 Special revolver firing handsamples, a substantial increase in the Pb and Sb averages for the indoor over the outdoor firings may have been caused by the wind encountered outdoors. The average Pb and Sb amounts from the .38 Special revolver outdoor firing handsamples (taken with tape immediately after firing) were too close to the handblank levels to be definitive in gunshot residue analysis, but the Pb and Sb indoor firing data showed levels substantially above the handblank levels. The fact that some of the ammunition used for the outdoor tests was not used in the indoor tests does not seem to influence this result. For Western (lupaloy bullet) cartridges, the averages for five outdoor firings were 0.57 μg Pb and 0.02 μg Sb, while the averages for nine indoor firings were 4.9 μg Pb and 0.16 μg Sb. For Remington (lead-nose bullet) cartridges, the averages for five outdoor firings were 0.92 μg Pb and 0.04 μg Sb, while the averages for six indoor firings were 2.8 μg Pb and 0.10 μg Sb. For 16 indoor firing handsamples of the .38 Special revolver, the Sb average of 0.13 μg is appreciable, but is less than the Gulf General Atomic 0.20- μg Sb average for 56 samples determined by means of neutron activation analysis.⁶

After comparing our .38 Special firing sample data and handblank data for Pb with the Gulf General Atomic data for Ba, we concluded that the combination of Pb and Sb analyses is better than the commonly used Ba and Sb combination. Of our 45 handblanks, 90 percent gave Pb amounts of 0.85 μg and below, while 100 percent of the sixteen .38 Special revolver, indoor, one-round firing handsamples gave more than 0.85 μg Pb. Of the 165 Class A

handblanks of the Gulf General Atomic study,* 90 percent gave Ba amounts of 0.34 μg and below, while 57 percent of the 56 .38 Special revolver one-round firing handsamples gave more than 0.34 μg Ba.⁶ Therefore, this comparison suggests that Pb detected by photoluminescence is a better indicator of residue than Ba as detected by neutron activation analysis.

In the limited data on the one-round, 9 mm pistol firing handsamples, Pb and Sb appear useful in distinguishing indoor firing samples from handblank levels but not useful for the outdoor firing samples.

A large, unexplainable discrepancy is observed in the results for firing the .380 caliber Browning semiautomatic between the 28 two-round, outdoor firing handsamples collected by water-wash and the six one-round, outdoor firing handsamples collected by tape. The average Pb amount for the one-round tape collection samples is twice that for the two-round water-wash samples reported in the earlier report on photoluminescence detection of gunshot residue.¹ The average Sb amount for the adhesive collection samples

*In the Gulf General Atomic work, four occupational groups were defined.

Class A included individuals with low exposure to Ba and Sb, and consisted of secretaries and laboratory technicians, among others. Class B was high in Ba and low in Sb, including plumbers, graphic artists, mechanics, draftsmen, and heating and air-conditioning repairmen. Class C was low in Ba but high in Sb, including electronic assemblers, in particular. Class D, high in both Ba and Sb, included auto mechanics, painters, machinists, and maintenance men.

is seven times that for the water-wash samples. A large range is observed in the Pb and Sb values for both indoor and outdoor firing samples.

Too little work was done with the .32 caliber revolver for discussion, but with the .32 caliber semiautomatic pistol the contrast between Sb and Pb levels in firing handsamples and in handblanks was apparent.

From the limited amount of .22 caliber revolver firings, it appears that, for the outdoor firing samples, Pb and Sb amounts often are indistinguishable from the handblank levels. The average Pb and Sb amounts in the case of indoor samples were a little greater than those for the outdoor samples. Of the seven indoor .22 caliber revolver firing samples, just two involved Sb-containing primers, but five of the seven samples gave Sb amounts of 0.03 μg or more. These results can be explained in terms of introduction of contamination from bullet lead or by residue retained from previous firings made with Sb-containing primers.

Insufficient work was done with the .22 caliber semiautomatic pistol to draw conclusions about the outdoor firings, but with the indoor firing handsamples the Pb and Sb data are, for the most part, useful in distinguishing firing samples from handblanks. As in the case of the .22 caliber revolver, Sb is detected in handsamples when the cartridge fired has an Sb-free primer. Although just two of the nine samples involved the use of cartridges with Sb-containing primers, all nine samples contained at least 0.03 μg Sb. The observation of antimony in residue from cartridges with antimony-free primers is not new.⁶ One possible source for this material is the small percentage of Sb with which the bullet lead is alloyed to harden it, and another is residue retained in the barrel of the gun from previous firings.

From our limited work to date, it would be difficult to make a comparison of the Pb and Sb levels in firing handsamples between the revolver and semi-automatic pistol firings. Only the .22 caliber handguns could be compared, and the data indicate the semiautomatic pistol leaves more gunshot residue on the firing hand than does the revolver.

B. Handblanks

Of our sampling of 45 handblanks, 33 subjects fall into the Class A category of the Gulf General Atomic work,⁶ and there are 12 subjects that fit into Class D of the same Gulf General Atomic study. In none of our 33 Class A handblanks was Sb detected (the minimum detectable limit being 0.01 μg Sb), while in the Gulf General Atomic study 40 percent of the Sb Class A handblank values were 0.01 μg or more. Only one of our 12 Class D handblanks (handsamples taken from painters, maintenance men, auto mechanics, and machinists) gave a detectable amount of Sb (0.03 μg), while in the Gulf General Atomic study, 59 percent of the Sb Class D handblanks were 0.03 μg or over. The apparent discrepancy between our Sb handblank data and those of Gulf General Atomic is not understood. Our Sb averages for handblanks and one-round, .38 Special revolver firing handsamples clearly were lower than the analogous Gulf General Atomic averages.

When one compares the average amount of Pb detected in handblanks collected using the water-wash and adhesive collection methods, it can be seen that about twice as much Pb was detected in the adhesive-collected samples as in the water-washings. Our 33 Class A handblanks collected with adhesive gave an average of 0.4 μg Pb, and the 20 water-wash-collected

specimens gave an average of 0.2 μg . Antimony was undetectable in both sets of handblanks.

C. Residue on Clothing

Data obtained from the pocket-residue analyses indicate that gunshot residue can be found readily in the pockets of shooters who have placed their hands in their pockets.* Clothing did not introduce luminescent interference in the analyses, and common levels of Sb in pockets in which no gunshot residue was present fell below the minimum detectable limit. Further work needs to be done to study the effect of time on the persistence of gunshot residue in pockets. It is likely that the persistence of residue transferred to a pocket by a hand, after shooting a gun, is longer than the persistence on the back of the firing hand itself. The examination of the handsamples taken from the firing hands after placing hands into pockets three times indicated that Sb could still be detected easily, although the amount may be down by a factor of six from the Sb amounts found immediately after firing. The Pb values obtained from the handsamples after placement into pockets and from the pockets themselves, were essentially indistinguishable from handblank Pb data. Only Sb appears to be useful in the persistence analysis. Our result showing that large amounts of Sb were removed from the firing hand by the placement of the hand in a pocket three times agrees with the findings of Kilty.⁴

*The mere presence of residue in a pocket would probably be less compelling evidence of a firing than, for example, the detection of large amounts of residue on the back of the firing hand taken together with small amounts of the nonfiring hand.

Nevertheless, detectable amounts of Sb remained on the hand. For seven .32 caliber semiautomatic pistol firing handsamples taken after subjects placed their hands into their pockets three times, on the average 0.12 μg of Sb was detected, whereas an average of 0.8 μg was observed immediately after firing. For four .45 caliber semiautomatic pistol firing handsamples, Kilty saw an Sb average of 0.19 μg , as compared to 1.3 μg immediately after firing. The amounts of Sb and Pb found on the firing handsample following placement of a hand into a pocket three times do not seem to be correlated with the amount of Sb and Pb found in the residue-containing pocket. In Table 2, for example, subjects 2 and 4 had roughly equal amounts of Sb in the pocket sample and in the handsample. Subject 5 had roughly equal amounts of Pb in the pocket sample and handsample. Subject 6 had much more Sb in the pocket sample than in the handsample, and subjects 3 and 6 had much more Pb in the pocket sample than in the handsample. Subjects 1, 3, 5, and 7 had more Sb in the handsample than in the pocket sample, and subjects 1, 2, 4, and 7 had more Pb in the handsample than in the pocket sample. These variations are consistent with the heterogeneous nature of the particulate residue deposit.

Tape samples collected from firing hands after one-round firings, followed by vigorous wiping for 15 sec in an attempt to remove all residue, showed that the Pb is usually reduced to the handblank level, and Sb is often reduced to a value close to or equal to the handblank level. In five of the seven handsamples, Sb was detected, but only two of those samples gave amounts over 0.03 μg . This result indicates the ability of a shooter to remove the residue from the shooting hand by deliberately and vigorously wiping it on

clothing. Further tests need to be done on this type of case, where both the clothing and the hands are sampled for Pb and Sb. Our Sb data on this 15-sec removal test are in agreement with the findings of Kilty.⁴ Our Sb average for seven firing handsamples was 0.04 μg , representing a 20-fold reduction, while Kilty's two firing samples taken after 15 sec of wiping hands on clothing showed an Sb amount of 0.05 μg ,⁴ representing a 26-fold reduction.

D. Persistence of Residue on Hands

In the studies of the persistence of gunshot residue with respect to time, when a .32 caliber semiautomatic pistol was used Pb on the hands was reduced to background levels at the 1-hr mark for both firing and nonfiring hands. The average Sb amounts for the 1-, 2-, 3-, and 4-hr persistence time intervals were 0.08, 0.06, 0.04, and 0.03 μg , respectively; therefore, Sb appears useful while Pb does not. When one compares the average amount of Sb in a handsample, taken immediately after firing a .32 caliber semiautomatic pistol, with the average amount of Sb 1 hr after firing, one sees an order-of-magnitude drop in the amount. Our results are in agreement with the findings of Kilty,⁴ which also showed a drop in Sb of about an order of magnitude from the 0-hr to the 1-hr samples. However, the residues produced by the semiautomatic pistols used in both of these studies may differ from those which would be obtained using other types of guns; this could influence the observed persistence, and some preliminary data from .38 caliber revolver firings suggest that this may indeed be the case. An order-of-magnitude drop for the average Pb amount (2.3 μg) on the firing handsample immediately after firing the .32 caliber semiautomatic pistol would

reduce the average Pb to below the average handblank level for Pb. This explains why the Pb levels seen at the 1-hr time interval are indistinguishable from the handblank level. On the nonfiring hand, Pb and Sb amounts in handsamples taken immediately after firing are indistinguishable from common handblank levels. Average Pb values at the 1-, 2-, 3-, and 4-hr time intervals after shooting are indistinguishable from the common Pb handblank levels, and no individual Pb value from the nonfiring handsamples was greater than 0.95 μg . Although no Sb was detected in any of the four handsamples from nonfiring hands taken immediately after the one-round, one-hand .32 caliber semiautomatic pistol firings, and although no Sb was detected in any of the four handsamples taken 4 hr after firing, Sb was detected in two of five nonfiring-hand handsamples taken for each of the 1-, 2-, and 3-hr time samplings after firing. This result suggests the transfer of residue from firing to nonfiring hand by activity over a 3-hr period after shooting. The findings of Kilty (using two subjects) also show that the Sb amounts increase on the nonfiring hand with time.

Most of the persistence data reported here are derived from firings of a .32 semiautomatic (Llama) and may not accurately reflect the persistence behavior of other types of guns and ammunition. A more definitive persistence study of a greater variety of residues is being undertaken, using both the luminescence and the particle analysis methods.

E. Adhesive Residue Collection

The tape adhesive sample collection method is rapid, and it can be used in conjunction with various techniques of analysis, such as inorganic luminescence, atomic absorption, chromatography, and scanning electron

microscopy. The examination of a tape sample with the SEM for particle identification can be followed immediately by the quantitative analysis of Sb and Pb using inorganic luminescence. The tape samples can be stored for an indefinite period of time without harm using a closed container to prevent contamination, and negligible Pb and Sb background interference is obtained from the tape in the photoluminescence analysis. The other hand-sampling techniques employed in gunshot residue collection exhibit inconveniences and drawbacks. The cotton swabs apply acid to the hand and in some instances have been contaminated with elements of interest to this determination. Paraffin lifts are inconvenient for field use, whereas cellulose acetate film lifts require high-purity material. With the water-wash collection method, which we previously employed, Sb determinations were hindered by background contamination, and the technique was inconvenient for field collection. With the tape method, these problems are minimized, and greater collection efficiency is observed. The tape method, which uses a common, commercially available adhesive material, can also be used to obtain spatial distribution information for gunshot residue on hands. Data obtained using the various collection techniques can be expected to differ quantitatively because nonidentical areas of the hand are sampled and efficiency of retention for various components of the residue can be expected to vary from method to method.

F. Filter Fluorimeter

The modified commercial filter fluorimeter provides the sensitivity required for Sb analysis. It was anticipated that detection based on excitation

at a single wavelength would be much less specific for Sb than measurements based on the entire excitation spectrum. However, the limited results for handsamples and handblanks indicate interfering signals from hand contaminants may not be a major problem for screening applications. For example, if a threshold of 0.03 μg Sb were used, none of the handblanks would have been interpreted to be false positive, and 77 percent of specimens collected immediately after firing would have been correctly judged positive. Although the instrument is limited to Sb detection, this may not be a serious restriction because Pb and Ba are much less valuable indicators of residue. The great advantage of the fluorimeter is its low cost (about \$800, plus about \$300 for auxiliary equipment) and the ease of operation. More extensive testing would be required before the feasibility of practical application could be assessed.

CHAPTER V. CONCLUSION

Inorganic luminescence analysis combined with the adhesive method of sample collection provides a convenient and effective method for elemental analysis of gunshot residue. Antimony appears to be the most useful indicator of the presence of residue because of its low abundance in handblanks. Results for handblanks have indicated significantly less Sb than has been reported in past surveys. This may be associated with the new collection and analysis procedures. If these findings are confirmed by subsequent independent studies, the utility of elemental analysis would have to be assessed more favorably than in the past for promptly collected residue and for delayed collection, provided the preliminary persistence data obtained thus far are typical.

Comparison of the new Pb data with prior studies of Ba in gunshot residue suggests that Pb is a more valuable measure of residue than Ba, which had been used in most previous detection work. However, both Pb and Ba have average handblank levels equivalent to the reduced average amounts of these elements that are found one hour after firing. Therefore, they are not expected to be useful indicators in typical casework involving live suspects. In cases of suicide or immediate collection of residue, they should provide a useful supplement to information from Sb.

It can be anticipated that overall detection performance would not be reduced excessively if analysis capability were limited to Sb. Considerable savings in equipment design could be achieved for a special purpose instrument limited to this function. However, over the long run, Pb will become more

useful as its environmental (automotive) source is eliminated, provided it is retained as a constituent of ammunition. Therefore, the future capability is sacrificed if a limited system is adopted.

Ability to detect residue transferred from hands to clothing pockets is a highly promising outcome of this study. A large part of the residue adheres to the cloth and can be removed by the adhesive technique. Since substantial persistence of this deposit is anticipated, it may become a valuable procedure. Also, transfer of residue from the firing to the nonfiring hand was observed consistently in tests of residue persistence, in agreement with results of other research groups.

NOTES

1. P. F. Jones and R. S. Nesbitt, "A Photoluminescence Technique for Detection of Gunshot Residue," Report No. ATR-74(7915)-1, El Segundo, Calif., The Aerospace Corp. (June 1974); also, Journal of Forensic Sciences, Vol. 20, 1975, pp. 231-242.
2. J. E. Wessel, P. F. Jones, Q. Y. Kwan, R. S. Nesbitt, and E. J. Rattin, "Gunshot Residue Detection: Survey and Assessment and Identification of Alternative Concepts," Report No. ATR-75(7915)-1, El Segundo, Calif., The Aerospace Corp. (September 1974).
3. R. S. Nesbitt, J. E. Wessel, and P. F. Jones, "Conclusive Detection of Gunshot Residue by the Use of Particle Analysis," Report No. ATR-75(7915)-2, El Segundo, Calif., The Aerospace Corp. (December 1974); also, submitted to Journal of Forensic Sciences.
4. J. W. Kilty, "Activity after shooting and its effect on the retention of primer residue," Journal of Forensic Sciences, Vol. 20, 1975, pp. 219-230.
5. V. P. Guinn, H. R. Lukens, and H. L. Schlesinger, "Applications of Neutron Activation Analysis in Scientific Crime Investigation," Report No. GA-9807, San Diego, Calif., Gulf General Atomic, Inc. (10 August 1970).
6. H. L. Schlesinger, H. R. Lukens, V. P. Guinn, R. P. Hackleman, and R. F. Korts, "Special Report on Gunshot Residues Measured by Neutron Activation Analysis," Report No. GA-9829, San Diego, Calif., Gulf General Atomic, Inc. (10 August 1970).

Table A1. .38 Special Smith and Wesson Revolver^a

A. Outdoor Firings			B. Indoor Firings		
Pb (µg)	Sb (µg)	Cartridge ^b	Pb (µg)	Sb (µg)	Cartridge ^b
0.55	- ^c	W, 158 gr LUB	3.5	0.12	W, 158 gr LUB
0.30	0.01	W, 158 gr LUB	5.1	0.17	W, 158 gr LUB
0.56	0.03	W, 158 gr LUB	4.7	0.12	W, 158 gr LUB
0.50	0.01	W, 158 gr LUB	5.0	0.09	W, 158 gr LUB
0.92	0.04	W, 158 gr LUB	7.8	0.25	W, 158 gr LUB
1.7	0.04	R, 158 gr RNL	5.3	0.18	W, 158 gr LUB
1.0	0.05	R, 158 gr RNL	1.7	0.07	W, 158 gr LUB
0.44	0.03	R, 158 gr RNL	4.8	0.30	W, 158 gr LUB
0.50	0.03	R, 158 gr RNL	5.9	0.18	W, 158 gr LUB
0.98	0.06	R, 158 gr RNL	2.3	0.08	R, 158 gr RNL
0.55	0.04	R, 125 gr JHP	2.1	0.14	R, 158 gr RNL
0.33	-	R, 125 gr JHP	2.2	0.07	R, 158 gr RNL
0.50	0.19	R, 125 gr JHP	1.7	0.07	R, 158 gr RNL
0.55	0.04	R, 125 gr JHP	3.9	0.11	R, 158 gr RNL
1.3	0.05	R, 125 gr JHP	4.4	0.13	R, 158 gr RNL
0.90	0.01	R, 125 gr JHP			
0.58	-	SV, 110 gr JHP			
0.40	0.06	SV, 110 gr JHP			
0.55	0.01	SV, 110 gr JHP			
0.43	0.02	SV, 110 gr JHP			
0.60	0.06	N, 158 gr JHP			
0.29	0.06	N, 158 gr JHP			
0.21	0.03	N, 158 gr JHP			
1.1	0.03	N, 158 gr JHP			
0.69	0.02	N, 158 gr JHP			
0.60	0.04	N, 158 gr RNL			
0.29	0.04	N, 158 gr RNL			
0.54	0.01	N, 158 gr RNL			
1.0	0.07	N, 158 gr RNL			
0.65	0.07	unk			
0.30	0.03	unk			

APPENDIX. HANDSAMPLE DATA

^aThe tape collection method of sampling was applied to the back of the firing hand following the one-round firing of a .38 Special Smith and Wesson revolver (2-in. barrel).

^bW = Western, R = Remington, SV = Super Vel, N = Norma, LUB = lubaloy bullet, RNL = lead round nose bullet, JHP = jacketed hollow point bullet, unk = unknown.

^cThis symbol indicates that no detectable amount was observed. For Sb, it indicates no evidence of Sb.

Table A2. 9 mm Browning Hi-Power Semiautomatic Pistol^a

A. Outdoor Firings			B. Indoor Firings		
Pb (µg)	Sb (µg)	Cartridge ^b	Pb (µg)	SB (µg)	Cartridge ^b
0.30	—	F, 123 gr mc	2.1	0.12	F, 123 gr mc
0.68	0.03	F, 123 gr mc	2.3	0.14	F, 123 gr mc
0.30	0.01	S, 100 gr JHP			
0.61	0.02	L, mc			
0.43	0.05	F, 123 gr mc			
1.1	0.02	F, 123 gr mc			
0.35	0.01	L, mc			
0.25	0.05	S, 100 gr JHP			
0.29	—	F, 123 gr mc			
Average:					
0.5	0.02				

^aThe tape collection method of sampling was applied to the back of the firing hand following the firing of one round from a 9 mm Browning Hi-Power semiautomatic pistol.

^bF = Federal, S = Speer, L = Lapua Patruunatehdas (Finnish), mc = metal case bullet, JHP = jacketed hollow point bullet.

Table A3. .380 Browning Semiautomatic Pistol^a

A. Outdoor Firing Samples ^b			B. Indoor Firing Samples		
Pb (µg)	Sb (µg)	Cartridge ^c	Pb (µg)	Sb (µg)	Cartridge ^c
1.0	0.22	R, 95 gr mc	1.1	0.33	R, 95 gr mc
3.6	0.40	W, mc	9.2	0.63	W, mc
4.2	0.90	W, mc	4.7	0.72	W, mc
2.8	0.90	W, mc	6.4	0.21	W, mc
0.90	0.08	R, 95 gr mc	3.2	0.54	W, mc
0.85	0.10	R, 95 gr mc			
Average			Average		
2.2	0.7		5.0	0.5	

^aThe tape collection method of sampling was applied to the back of the firing hand following the one-round firing of a .380 Browning semiautomatic pistol.

^bTwenty-eight handsamples from two-round outdoor firing collected by a water wash gave Pb and Sb averages of 1.1 and 0.09 µg, respectively.

^cR = Remington, W = Western, mc = metal case bullet.

Table A5. .32 Llama Semiautomatic Pistol^a

Indoor Firings					
Pb (μg)	Sb (μg)	Cartridge ^b	Pb (μg)	Sb (μg)	Cartridge ^b
3.8	1.7	F, 71 gr mc	0.9	0.19	F, 71 gr mc
1.7	0.64	F, 71 gr mc	1.9	0.98	F, 71 gr mc
1.8	0.83	F, 71 gr mc	1.8	0.91	F, 71 gr mc
2.4	0.66	F, 71 gr mc	1.7	0.79	F, 71 gr mc
0.8	0.07	F, 71 gr mc	1.5	0.23	F, 71 gr mc
1.8	0.30	F, 71 gr mc	5.0	1.7	F, 71 gr mc
1.5	0.35	W, mc	3.6	1.7	F, 71 gr mc
2.1	0.28	W, mc	3.8	1.3	F, 71 gr mc
0.7	0.11	R, 71 gr mc	3.7	0.95	F, 71 gr mc
1.4	0.12	F, 71 gr mc	5.0	1.8	F, 71 gr mc
			Average		
			2.3	0.8	

^aThe tape collection method of sampling was applied to the back of the firing hand following the one-round firing of a .32 Llama semiautomatic pistol.

^bF = Federal, W = Western, R = Remington, mc = metal case bullet.

Table A6. .22 Harrington and Richardson Revolver^a

A. Outdoor Firings			B. Indoor Firings		
Pb (μg)	Sb (μg)	Ammo Brand	Pb (μg)	Sb (μg)	Ammo Brand
2.9	0.12	Western Super X	1.6	0.13	Western Super X
1.8	0.07	Western Super X	0.91	0.03	Federal
2.6	0.09	Western Super X	2.1	0.08	Federal
0.43	-	Western Super X	1.7	0.04	Browning
0.26	-	Western Super X	0.7	0.03	Browning
0.53	-	Western Super X	0.6	-	Browning
0.69	0.10	Federal	2.0	0.01	Remington
0.50	-	Federal	4.0 ^b	0.5 ^b	Remington
0.35	0.01	Federal			
0.76	0.04	Federal			
1.0	-	Remington			
1.0	0.01	Remington			
0.22	-	Remington			
Average			Average		
1.0	0.03		1.4	0.05	

^aThe tape collection method of sampling was applied to the back of the firing hand following the one-round firing of a .22 Model 929 Harrington and Richardson revolver (2-in. barrel). Copper-coated ammunition was used.

^bThis result is unusual since no Sb is contained in the cartridge primer in this case. The Pb and Sb entries for this sample are omitted in the calculation of the averages for Pb and Sb.

Table A7. .22 Hi-Standard Semiautomatic Pistol^a

A. Outdoor Firings			B. Indoor Firings		
Pb (μg)	Sb (μg)	Ammo Brand	Pb (μg)	Sb (μg)	Ammo Brand
0.5	0.02	Browning	1.4	0.03	Western Super X
0.5	0.03	Federal	2.4	0.09	Federal
			2.1	0.07	Federal
			3.0	0.09	Browning
			1.3	0.04	Browning
			1.2	0.04	Remington
			3.1	0.05	CCI
			1.6	0.14	CCI
			1.0	0.07	CCI
			Average		
			1.9	0.07	

^aThe tape collection method of sampling was applied to the back of the firing hand following the one-round firing of a .22 model M-101 Hi-Standard semiautomatic pistol. Copper-coated ammunition was used.

Table A8. Handblanks^a

Occupation	Pb (μg)	Sb (μg)	Occupation	Pb (μg)	Sb (μg)
Laboratory worker	0.30	-	Carpet layer	0.55	-
Laboratory worker	0.26	-	Typist	0.12	-
Laboratory worker	0.55	-	Typist	0.11	-
Laboratory worker	0.60	-	Liquor store clerk	0.17	-
Laboratory worker	0.25	-	Barber	0.18	-
Laboratory worker	0.25	-	Pharmacist	0.08	-
Laboratory worker	0.45	-	Office worker	0.10	-
Laboratory worker	0.45	-	Teacher	0.10	-
Laboratory worker	0.25	-	Disabled	0.17	-
Laboratory worker	0.38	-	High school student	0.19	-
Laboratory worker	0.12	-	Painter	0.86	-
Laboratory worker	0.37	-	Painter	0.24	-
Laboratory worker	0.60	-	Machinist	0.76	-
Laboratory worker	0.20	-	Machinist	0.70	-
Laboratory worker	0.40	-	Machinist	0.34	-
Laboratory worker	0.30	-	Machinist	0.60	0.03
Laboratory worker	0.90	-	Machinist	0.25	-
Laboratory worker	0.85	-	Auto mechanic	0.62	-
Laboratory worker	0.95	-	Auto mechanic	1.3	-
Laboratory worker	0.53	-	Maintenance man	0.70	-
Floor sander	0.34	-	Maintenance man	0.43	-
TV repairman	0.34	-	Maintenance man	0.63	-
Bicycle factory worker	0.20	-			

^aThe tape collection method was used to remove debris from the back of the right hand of persons who had not recently fired a gun.

Table A9. Effect of Time on Persistence of Gunshot Residue^a

Subject	Time Period Between Firing and Collection (hr)	Firing Hand		Nonfiring Hand		
		Pb (µg)	Sb (µg)	Pb (µg)	Sb (µg)	
1	0	0.88	0.19	0.42	-	
2	0	1.9	0.98	0.22	-	
4	0	3.7	0.95	0.75	-	
6	0	5.0	1.8	0.60	-	
		Average	2.9	1.0	0.5	-
1	1	0.85	0.12	0.79	0.03	
2	1	0.40	0.05	0.13	-	
3	1	0.72	0.07	0.95	0.03	
6	1	0.25	0.03	0.15	-	
8	1	0.63	0.12	0.53	-	
		Average	0.6	0.08	0.5	0.01
1	2	1.3	0.15	0.62	0.03	
2	2	0.48	0.12	0.13	-	
3	2	0.33	-	0.33	-	
4	2	1.3	0.03	0.95	0.02	
8	2	0.31	-	0.30	-	
		Average	0.8	0.06	0.5	0.01
1	3	1.2	0.04	0.70	0.02	
2	3	0.48	0.06	0.42	-	
3	3	1.3	0.07	0.72	0.01	
4	3	0.69	0.03	0.78	-	
9	3	0.23	-	0.16	-	
		Average	0.8	0.04	0.6	0.01
1	4	0.80	0.03	0.53	-	
1	4	1.0	0.07	0.60	-	
2	4	0.51	0.02	0.30	-	
9	4	0.25	-	0.30	-	
		Average	0.6	0.03	0.43	-

^aFollowing the firing of one round with one hand from a .32 Lama semiautomatic pistol, 0-, 1-, 2-, 3-, and 4-hr intervals were allowed to pass before sampling of the hands with the tape collection method. The only restriction on the activity of the hands over these time intervals was that the subject did not wash them.

GLOSSARY

ACP	Automatic Colt Pistol: used together with caliber designation to describe a specific cartridge configuration
Ba	barium
cm	centimeter
Dewar	vacuum flask, similar to a thermos bottle, that provides a high degree of thermal insulation for the content, which is commonly a cryogenic fluid such as liquid nitrogen
g	gram
gr	grain: bullet weight is given in grains; 1 gr = 0.0648 grams
Handblank	specimen taken from the back of the hand of a person who has not fired a gun
Handsample	specimen of gunshot residue taken from the back of the hand of a person who may have fired a gun
HCl	hydrochloric acid
K	Kelvin
ml	milliliter
mm	millimeter
Monochromator	wavelength selector for the ultraviolet, visible, or infrared spectrum consisting of mirrors and a grating or prism

nm nanometer: a unit of length equal to 10^{-7} cm that is commonly used in the measurement of light wavelength

Pb lead

Photoluminescence light emitted by a chemical species in the visible or ultraviolet wavelength region of the electromagnetic spectrum when the species has been excited with radiation of higher energy

Photomultiplier an extremely sensitive instrument, which is used for the detection of light, that consists of a photocathode, an anode, and a series of electron multiplier dynodes

Sb antimony

μ g microgram: one millionth of a gram (10^{-6} g)

END