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Evaluation of a Photoluminescence Technique for the Detection of Gunshot Residue

This is the second paper in a series describing the photoluminescence technique for detection of gunshot residue. It presents results of a substantial number of analyses of samples obtained for a variety of handguns and cartridges and collected under various conditions. A primary objective of the second study has been to evaluate factors which affect the utility of the method, such as the persistence of residue on the hand after firing, the effect of activity on retention of residue, and the possible presence of identifiable residue on surfaces other than the hand. The data represent an extensive addition to existing information on lead in gunshot residue and samples from several environmental conditions. A new method of residue collection, based on an adhesive layer, was used to collect samples from hands. It was found to be superior to the wash procedure used in the previously reported work [1]. For background information on the luminescence method the reader is referred to the earlier report.

Procedure

After a gun was fired, the back of each hand was sampled separately with a 2.5-cm diameter disk that was coated on one side with Scotch No. 465 adhesive transfer tape. The disk was initially pressed against the web area and then repeatedly pressed against the outside edge of the forefinger, the inside edge of the thumb, and then the back of the hand until the adhesive lost sticking ability. To prepare solutions for lead (Pb) and antimony (Sb) analysis, 0.5 ml of 7*M* 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 disks that were used to support the adhesive. After the HCl reaction, the acid sample was placed in a Suprasil quartz sample tube of precision bore, 4-mm inside diameter and 6-mm outside diameter.² The tube was immersed into liquid nitrogen in a quartz optical Dewar flask and was analyzed for Pb and Sb content. Acceptably effective residue collection was demonstrated in that suc-

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² 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 Dewar flasks, must have low fluorescence.

cessive samplings of a firing hand with fresh adhesive blocks yielded less than 50% additional residue.

The photoluminescence instrumentation consisted of an excitation system and emission monitor (light detection) system. Excitation light from a quartz-xenon arc lamp was passed through a scanning monochromator and imaged on the sample Dewar flask. Luminescence from the sample was focused onto the slits of an emission monochromator 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 a Ga-As cathode photomultiplier, which has a flat spectral response from 400 to 700 nm. The photomultiplier signal was amplified and the spectra recorded with an X-Y recorder.

For Sb analysis, the detection 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 cutoff filter of low fluorescence, Schott FG-10, was placed between the sample compartment and the emission monochromator 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 filters were not required. Typical analysis data are shown in Fig. 1.

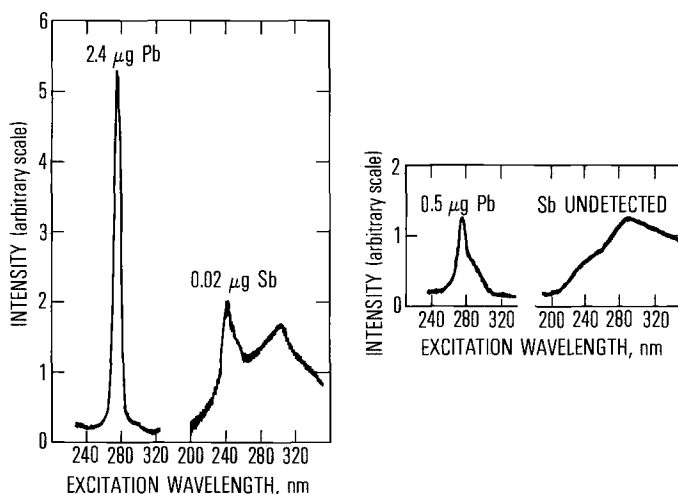


FIG. 1—Representative low-temperature luminescence excitation spectra for Pb and antimony of samples: (left) after firing one round from a .22 revolver and (right) a handblank. Antimony emission was monitored at 660 nm while the excitation was scanned, and Pb emission was monitored at 385 nm during the excitation scan.

The guns used in this study were a .22-caliber Harrington and Richardson Model 929 revolver (2-in. or 51-mm barrel), a .22-caliber Hi-Standard Model M-101 pistol, a .32-caliber revolver of unknown make and very poor condition, a .32-caliber Llama pistol, a .380 Browning pistol, a 9-mm Browning Hi-Power pistol, and a .38 Special Smith & Wesson revolver (2-in. or 51-mm barrel). All except the .32 revolver were in good condition. All of the 168 test firings were single-round firings using commercial brand ammunition, as specified in the tables of results. 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.

Results

Handblanks

Using the tape collection method, samples were taken from 45 different people including laboratory workers, painters, machinists, auto mechanics, maintenance men, and workers in other fields, while at work. These samples from people who had not fired a weapon are termed "handblanks" in this paper. Detectable Sb (amounts equal to and greater than $0.01 \mu\text{g}$) was observed in just one tape-lift handblank ($0.03 \mu\text{g}$), and the Pb average was $0.4 \mu\text{g}$. Table 1 and Fig. 2 present the amounts of Pb and Sb found. Just one Pb value above $1.0 \mu\text{g}$ was seen.

TABLE 1—Summary of data for samples collected immediately after firing and for handblanks.

Sample	Outdoor Firings			Indoor Firings		
	Avg Pb, μg	Avg Sb, μg	Samples, <i>n</i>	Avg Pb, μg	Avg Sb, μg	Samples, <i>n</i>
Revolvers						
.38	0.64 ± 0.33	0.04 ± 0.04	31 ^a	4.0 ± 1.8	0.14 ± 0.07	15 ^h
.32	1.3	0.05	3 ^b	3.0	0.07	2 ^b
.22	1.0 ± 0.9	0.03 ± 0.05	13 ^c	1.4 ± 0.6	0.05 ± 0.05	6 ⁱ
Pistols						
9 mm	0.5 ± 0.3	0.02 ± 0.02	9 ^d	2.2	0.1	2 ^j
.380	2.2 ± 1.5	0.4 ± 0.4	6 ^e	5.0 ± 3.1	0.5 ± 0.2	5 ^k
.32	2.3 ± 1.3	0.8 ± 0.6	20 ^l
.22	0.5	0.03	2 ^f	1.9 ± 0.8	0.07 ± 0.03	9 ^m
Handblanks	0.4	<0.01	45 ⁿ

^a Smith & Wesson .38 Special revolver, 2-in. (51-mm) barrel; Western Lubaloy 158-grain, Remington 158-grain round nose lead, Super Vel 110-grain jacketed hollow point, and Norma 158-grain round nose lead ammunition.

^b Four-inch (102-mm) barrel, .32 revolver of unknown make, poor condition; Western Lubaloy ammunition.

^c Model 929 Harrington and Richardson .22 revolver, 2-in. (51-mm) barrel; 40-grain Western Super X Lubaloy, Federal, and Remington copper-coated ammunition.

^d Browning 9-mm High Power pistol; Federal 123-grain full metal jacket, Speer 100-grain jacketed hollow point, and Lapua full metal jacket ammunition.

^e Browning .380 pistol; Remington 95-grain metal jacket and Western metal jacket ammunition.

^f Hi-Standard .22 pistol, Browning and Federal ammunition.

^g Obtained from 45 individuals representing diverse occupational groups.

^h Smith & Wesson .38 Special revolver, 2-in. (51-mm) barrel; Western 158-grain Lubaloy and Remington 158-grain round nose lead ammunition.

ⁱ Harrington and Richardson .22 revolver, 2-in. (51-mm) barrel; Western Super X Lubaloy, Federal, Browning, and Remington copper-coated ammunition.

^j Browning 9-mm High Power pistol; Federal 123-grain metal jacket ammunition.

^k Browning .380 pistol; Remington and Western 95-grain metal jacket ammunition.

^l Llama .32 pistol; Federal and Western 71-grain metal jacket ammunition.

^m Hi-Standard Model M-101 .22 pistol; Western Super X Lubaloy, Federal, Browning, Remington, and C.C.I. copper-coated ammunition.

Samples Collected Immediately After Firing

A summary of the average amounts of Pb and Sb collected immediately after one-round firings using the adhesive layer sampling procedure is presented in Table 1. More detailed data, including specifications for the ammunition used, are given in Tables 2 through 8. Because indoor and outdoor firings can involve substantial differences in ambient conditions, data have been reported separately for the two situations. Wind velocity was not monitored but was normally low. Indoor firings were conducted from partially enclosed shooting booths in a large firing range. Figures 3 to 6 are histograms of analysis results for the .38 revolver and the .32 Llama pistol. Arrows in the figures denote the approximate upper range of the amount commonly found in handblanks.

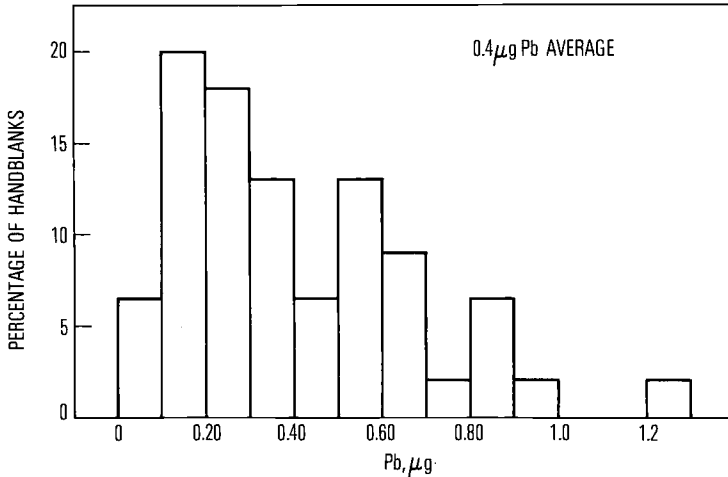


FIG. 2—Lead found in handblanks taken with the tape collection method.

Considerably more residue was found indoors than outdoors, and for the .38 revolver firings, all indoor samples had more Pb and Sb than any of the handblanks. With the .32 Llama pistol, all analyses for antimony exceeded the $0.03 \mu\text{g}$ obtained for the highest handblank. For Pb, 85% of firing samples exceeded the $1.3 \mu\text{g}$ obtained for the maximum handblank analysis. In the case of the .22 revolver and pistol, most results were only slightly above handblank levels. It was noted that Sb was detected in samples obtained for firings of Remington and Winchester-Western ammunition, which do not contain Sb in the primer.

Persistence of Residue in Firing Samples

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, following lines suggested by the original work of Kilty [2]. The major tests were carried out with one-round, one-hand, indoor firings of a .32 Llama pistol using Federal brand ammunition (71-grain, fully jacketed bullets).

In the first persistence experiment, one round was fired with the .32 pistol and then the firing hand was placed 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 9 lists the data. The pocket into which the firing hand was placed three times was also sampled, and Table 9 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. Tape adhesive mounted on 1-in. diameter sampling disks was pressed against the inside lip of the pockets to collect the residue.

One-round, one-hand, indoor firings with the same ammunition was conducted with the .32 Llama pistol, followed by vigorous wiping of the hands on clothing for about 15 s to remove the residue from the hands. The average amounts of Pb and Sb found on the firing samples after this activity were 0.6 and $0.4 \mu\text{g}$, respectively. The data for the seven samples analyzed are listed in Table 10.

Tests of the effect of normal activity on persistence of residue were made for one-

TABLE 2—.38 Special Smith & Wesson revolver.^a

Outdoor Firings			Indoor Firings		
Pb, μ g	Sb, μ g	Cartridge ^b	Pb, μ g	Sb, μ g	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	... ^c	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	... ^c	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	unknown
0.30	0.03	unknown

^aThe tape collection method of sampling was applied to the back of the firing hand after firing one round from a .38 Special Smith & Wesson revolver (2-in. or 51-mm barrel).

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

^cNo detectable amount was observed.

hand, indoor firings of the .32 pistol, which were followed by unrestricted activity for specified periods of time (except that washing the hands was forbidden). The backs of both hands were sampled with tape following 1, 2, 3, and 4-h time delays. Figure 7 shows the average amounts of Sb found on the samples after the different time periods. The largest declines in amounts of Sb were seen between the immediate and 1-h samplings. For the 1-h time interval and longer, the Pb levels were indistinguishable from background levels. Samples from the nonfiring hands gave undetectable levels at the 0 and 4-h intervals, while for each of the 1, 2, and 3-h time intervals after firings two of the five samples contained detectable Sb.

Data on Pb and Sb from limited persistence tests using several different guns and makes of ammunition are shown in Tables 11 and 12. During the interval between the firing (one round with one hand, indoors) and sample collection, unrestricted activity was allowed except that hands were not washed.

TABLE 3—Outdoor firing of 9-mm Browning pistol.^a

Pb, μg^b	Sb, μg^c	Cartridge ^d
0.30	...	F, 123 gr FMJ
0.68	0.03	F, 123 gr FMJ
0.30	0.01	S, 100 gr JHP
0.61	0.02	L, FMJ
0.43	0.05	F, 123 gr FMJ
1.1	0.02	F, 123 gr FMJ
0.35	0.01	L, FMJ
0.25	0.05	S, 100 gr JHP
0.29	...	F, 123 gr FMJ

^a The tape collection method of sampling was applied to the back of the firing hand after firing one round from a 9-mm Browning Hi-Power pistol. For indoor firings with Federal 123-grain fully jacketed cartridges, Pb was 2.1 and 2.3 μg and Sb was 0.12 and 0.14 μg .

^b Average, 0.5 μg ; standard deviation, $\pm 0.3 \mu\text{g}$.

^c Average, 0.02 μg ; standard deviation, $\pm 0.02 \mu\text{g}$.

^d F = Federal, S = Speer, L = Lapua (Finnish), FMJ = full metal jacket, and JHP = jacketed hollow point cartridge.

TABLE 4—.380 Browning pistol.^a

Outdoor Firing Samples ^b			Indoor Firing Samples		
Pb, μg^c	Sb, μg^d	Cartridge ^e	Pb, μg^f	Sb, μg^g	Cartridge ^e
1.0	0.22	Remington	1.1	0.33	Remington
3.6	0.40	Western	9.2	0.63	Western
4.2	0.90	Western	4.7	0.72	Western
2.8	0.90	Western	6.4	0.21	Western
0.90	0.08	Remington	3.2	0.54	Remington
0.85	0.10	Remington

^a The tape collection method of sampling was applied to the back of the firing hand after firing one round from a .380 Browning pistol.

^b Twenty-eight samples from two-round outdoor firings collected by washing with water gave Pb and Sb averages of 1.1 and 0.09 μg , respectively.

^c Average, 2.2 μg ; standard deviation, $\pm 1.5 \mu\text{g}$.

^d Average, 0.4 μg ; standard deviation, $\pm 0.4 \mu\text{g}$.

^e All were 95-grain, round-nose, fully jacketed cartridges.

^f Average, 5.0 μg ; standard deviation, $\pm 3.1 \mu\text{g}$.

^g Average, 0.5 μg ; standard deviation, $\pm 0.2 \mu\text{g}$.

Discussion

Handblanks

Of our sampling of 45 handblanks, 33 subjects fall into the Class A³ category of the Gulf General Atomic Work [3], and there are 12 subjects that fit into Class D of the

³ 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.

TABLE 5—.32 revolver.^a

Outdoor Firings		Indoor Firings	
Pb, μg^b	Sb, μg^c	Pb, μg	Sb, μg
1.9	0.03	2.7	0.05
0.7	0.05	3.5	0.08
1.3	0.07

^a The tape collection method of sampling was applied to the back of the firing hand after firing one round from a .32 revolver (4-in. or 102-mm barrel) of unknown make and poor mechanical condition. The cartridge used in each test was of Western manufacture with Lubaloy bullet.

^b Average, 1.3 μg ; standard deviation, $\pm 0.6 \mu\text{g}$.

^c Average, 0.05 μg ; standard deviation, $\pm 0.02 \mu\text{g}$.

TABLE 6—Indoor firings of a .32 Llama pistol.^a

Pb, μg	Sb, μg	Cartridge ^b	Pb, μg^c	Sb, μg^d	Cartridge ^b
3.8	1.7	Federal	0.9	0.19	Federal
1.7	0.64	Federal	1.9	0.98	Federal
1.8	0.83	Federal	1.8	0.91	Federal
2.4	0.66	Federal	1.7	0.79	Federal
0.8	0.07	Federal	1.5	0.23	Federal
1.8	0.30	Federal	5.0	1.7	Federal
1.5	0.35	Western	3.6	1.7	Federal
2.1	0.28	Western	3.8	1.3	Federal
0.7	0.11	Remington	3.7	0.95	Federal
1.4	0.12	Federal	5.0	1.8	Federal

^a The tape collection method of sampling was applied to the back of the firing hand after firing one round from a .32 Llama pistol.

^b All were 71-grain, round-nose, fully jacketed cartridges.

^c Average, 2.3 μg ; standard deviation, $\pm 1.3 \mu\text{g}$.

^d Average, 0.8 μg ; standard deviation, $\pm 0.6 \mu\text{g}$.

same 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% of the Sb Class A handblank values were 0.01 μg or more. Only 1 of our 12 Class D handblanks (samples 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% 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 revolver firing samples clearly were lower than the analogous Gulf General Atomic averages. This may imply that the photoluminescence method combined with tape collection provides a response more specific to residue than that obtained in the previous neutron activation analysis work. If so, lower Sb threshold levels would be appropriate. These results are listed in Table 13.

Samples Collected Immediately After Firing

In the .38 revolver firing samples, the conditions encountered outdoors may have caused the Pb and Sb averages to be lower than those measured for indoor firings. The

TABLE 7—.22 Harrington and Richardson revolver^a.

Outdoor Firings			Indoor Firings		
Pb, μg^b	Sb, μg^c	Cartridge	Pb, μg^d	Sb, μg^e	Cartridge
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 ^f	0.5 ^f	Remington
0.35	0.01	Federal
0.76	0.04	Federal
1.0	...	Remington
1.0	0.01	Remington
0.22	...	Remington

^a The tape collection method of sampling was applied to the back of the firing hand after firing one round from a .22 Model 929 Harrington and Richardson revolver (2-in. or 51-mm barrel). Copper-coated ammunition was used.

^b Average, 1.0 μg ; standard deviation, $\pm 0.9 \mu\text{g}$.

^c Average, 0.03 μg ; standard deviation, $\pm 0.05 \mu\text{g}$.

^d Average, 1.4 μg ; standard deviation, $\pm 0.6 \mu\text{g}$.

^e Average, 0.05 μg ; standard deviation, $\pm 0.05 \mu\text{g}$.

^f This result is unusual because 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.

TABLE 8—Indoor firing of .22 Hi-Standard pistol.^a

Pb, μg^b	Sb, μg^c	Cartridge
1.4	0.03	Western Super X
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	C.C.I.
1.6	0.14	C.C.I.
1.0	0.07	C.C.I.

^a The tape collection method of sampling was applied to the back of the firing hand after firing one round from a .22 model M-101 Hi-Standard pistol. Copper-coated ammunition was used. For outdoor firings, with Browning ammunition, Pb was 0.5 μg and Sb was 0.02 μg ; with Federal ammunition, Pb was 0.5 μg and Sb was 0.3 μg .

^b Average, 1.9 μg ; standard deviation, $\pm 0.8 \mu\text{g}$.

^c Average, 0.07 μg ; standard deviation, $\pm 0.03 \mu\text{g}$.

average Pb and Sb amounts from the .38 revolver outdoor firing samples (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. For Western (Lubaloy[®]) 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 round-nose lead cartridges, the averages for the five outdoor firings were 0.92 μg Pb and 0.04 μg Sb, while the averages for six in-

TABLE 9—Removal of gunshot residue by placing hands in pockets.

Samples of Firing Hands After Placement into Pocket Three Times ^a			Samples Taken From Pockets After Firing Hand Was Placed Inside Pocket Three Times ^b			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	... ^c
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	... ^c
7	1.7	0.18	7	0.47	0.12	7	0.17	... ^c
Average	0.9	0.12	Average	0.8	0.10	Average	0.28	...
Standard deviation	± 0.5	± 0.06	Standard deviation	± 0.5	± 0.02	Standard deviation	± 0.27	...

^a Following the firing with one hand of one round from a .32 caliber Llama 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 collection method. The average amounts of Pb and Sb found immediately after firing were 2.3 and 0.8 μg , respectively.

^b Following the firing with one hand of one round from a .32 caliber Llama 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 collection method. The main area of the pocket sampled was the inside of the lip.

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

TABLE 10—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	0.67	0.04
Standard deviation	± 0.36	± 0.04

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

TABLE 11—Effect of time on persistence of gunshot residue studied with a variety of guns.

Time, h	Handgun Used	Cartridge	Pb, μg ^a	Sb, μg
1	.38 Special Smith & Wesson revolver	Remington	1.1	0.05
1	.38 Special Smith & 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 pistol	Remington	2.0	0.08
2	.22 Hi-Standard pistol	Western Super X	1.1	0.01
2	.22 Hi-Standard pistol	Federal	1.5	0.03
2	.22 Smith & Wesson revolver	Browning	0.6	...
2	.38 Special Smith & Wesson revolver	Norma	3.0	0.13
2	.38 Special Smith & Wesson revolver	Norma	2.9	0.09

TABLE 12—Effect of time on persistence of gunshot residue. ^a

Subject	Time Between Firing and Collection, h	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	...

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

door firings were 2.8 μg Pb and 0.10 μg Sb. For 15 indoor firing samples of the .38 revolver, the Sb average of 0.14 μg is appreciable but is less than the Gulf General Atomic average of 0.20 μg Sb for 56 samples determined by means of neutron activation analysis [3].

After comparing our .38 firing sample data and handblank data for Pb with the Gulf General Atomic data for barium (Ba), we conclude that the combination of Pb and Sb analyses is better than the commonly used Ba and Sb combination. Of our 45 handblanks, 90% gave Pb amounts of 0.85 μg and below, while 100% of the sixteen .38 revolver, indoor, one-round firing samples gave more than 0.85 μg Pb. Of the 165 Class A handblanks of the Gulf General Atomic study, 90% gave Ba amounts of 0.34 μg and below, while 57% of the 56 .38 revolver one-round firing samples gave more than 0.34 μg Ba [3]. Therefore, this comparison suggests that Pb detected by photoluminescence is a better indicator of residue than Ba as detected by neutron activation analysis.

A large, unexplained discrepancy is observed between results for firing the .380 Browning reported in the earlier report on photoluminescence detection of gunshot residue and this more recent study. In the earlier work, 28 two-round, outdoor firing samples were collected by a wash with water and are to be compared with the 6 one-round, outdoor firing samples collected by tape in this study. The average Pb amount for the one-

TABLE 13—*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	...			

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

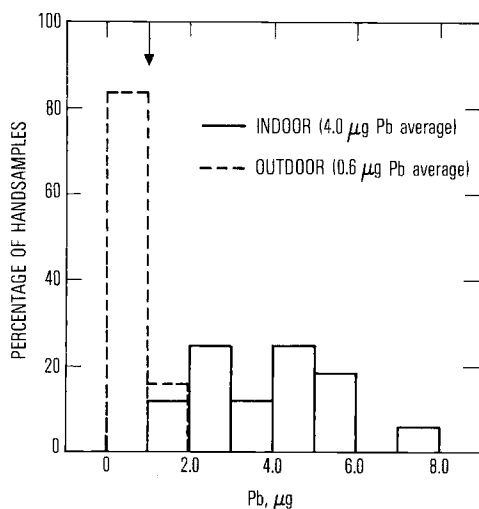


FIG. 3—Lead found in samples from indoor and outdoor firings of one round from a .38 Special Smith & Wesson revolver. Levels for 44 of the 45 handblanks (arrow) were below 1 μg . Samples were collected with tape. The vertical coordinate refers to the percentage of results falling within the range of the horizontal coordinate.

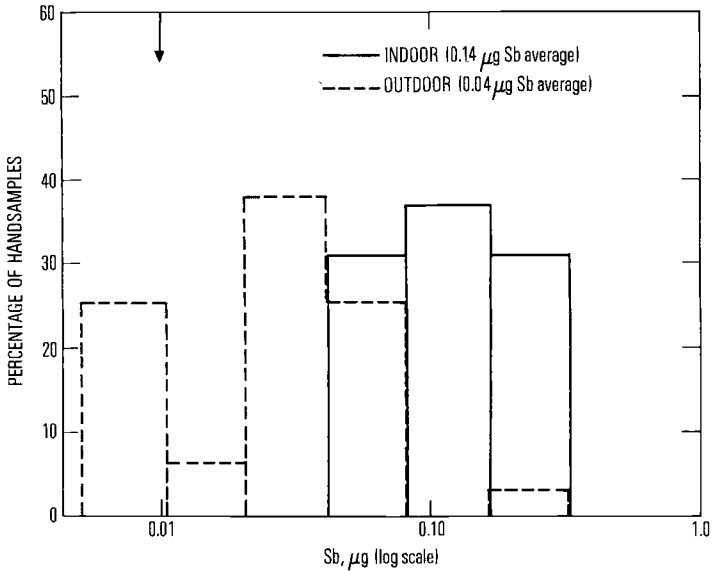


FIG. 4—Antimony found in samples from indoor and outdoor firings of one round from a .38 Special Smith & Wesson revolver. Levels for 44 of the 45 handblanks (arrow) were below 0.01 μg . Samples were collected with tape. The logarithmic scale of the horizontal coordinate shows the separation of indoor and outdoor firings more clearly than a linear scale would.

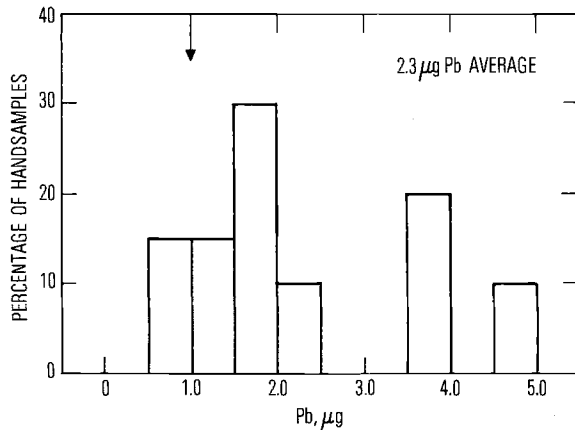


FIG. 5—Lead found in samples from indoor firing of one round from a .32 Llama pistol. Levels for 44 of the 45 handblanks (arrow) were below 1.0 μg . Samples were collected with tape.

round, tape collection samples is twice that for the two-round, water-wash samples reported earlier. The average Sb amount for the adhesive collection samples is four 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 revolver for discussion, but with the .32 pistol the contrast between Sb and Pb levels in firing samples and in handblanks was apparent.

From the limited amount of .22 revolver firings, it appears that for the outdoor firing

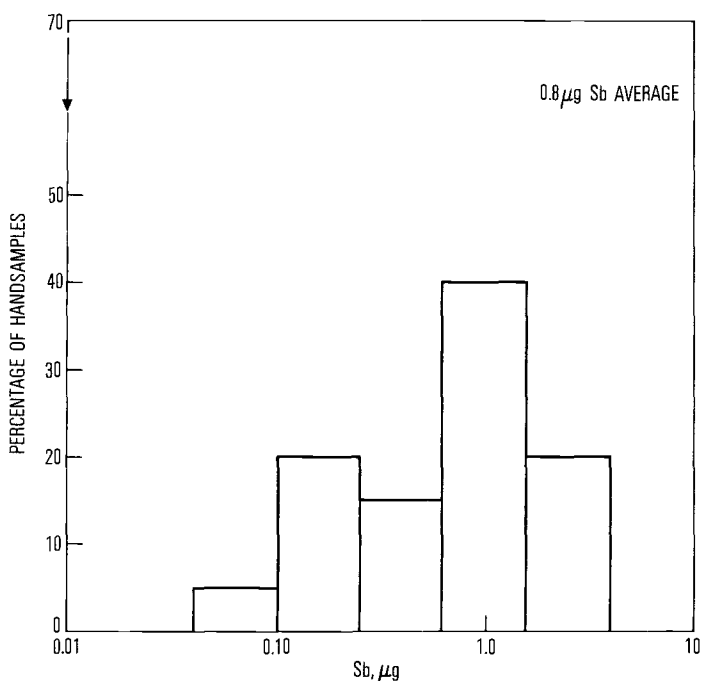


FIG. 6—Antimony found in samples from indoor firing of one round from a .32 Llama pistol. Levels for 44 of the 45 handblanks (arrow) were below 0.01 μg . Samples were collected with tape. The logarithmic scale for amounts of Sb emphasizes the separation of firing samples from the handblanks.

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 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 pistol to draw conclusions about the outdoor firings, but with the indoor firing samples the Pb and Sb data are, for the most part, useful in distinguishing firing samples from handblanks. As in the case of the .22 revolver, Sb is detected in samples 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 [3].

Residue on Clothing

Data obtained from the pocket-residue analyses indicate that gunshot residue can be found readily in the pockets of firers who have placed their hands in their pocket.⁴ Clothing did not introduce interference from luminescence in the analyses, and common

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 a small amount on the nonfiring hand.

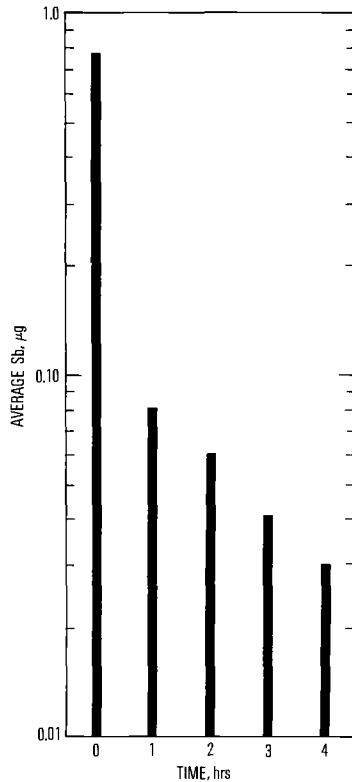


FIG. 7—Persistence with time of Sb on firing hand after firing one round from a .32 Llama pistol. Samples were collected with tape. Four firings were made for the 0 and 4-h delay periods; five firings were used for the 1, 2, and 3-h delays.

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 samples 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 hands after placement into pockets themselves were essentially indistinguishable from handblank Pb data. Only Sb appears to be useful in the persistence analysis. Our results 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 [2]. Nevertheless, detectable amounts of Sb remained on the hand. For seven .32 pistol firing samples taken after subjects placed their hands into their pockets three times an average of $0.12 \mu\text{g}$ of Sb was detected, whereas an average of $0.8 \mu\text{g}$ was observed immediately after firing. For four .45 pistol firing samples, Kilty saw an Sb average of $0.19 \mu\text{g}$ as compared to $1.3 \mu\text{g}$ immediately after firing. The amounts of Sb and Pb found on the firing sample following placement of a hand into a pocket three times are not correlated with the amount of Sb and Pb found in the residue-containing pocket. These variations in ratios of Sb to Pb, shown in Table 9, are consistent with the heterogeneous nature of the particulate residue deposit.

Tape samples for one-round firings that were collected from hands that had been vigorously wiped for 15 s 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 samples Sb was detected, but only two of those samples gave amounts over 0.03 μg . This result indicates the ability of a firer 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.

Persistence of Residue on Hands

In the studies of the persistence of gunshot residue with respect to time, when a .32 pistol was used, Pb on the hands was reduced to background levels at the 1-h mark for both firing and nonfiring hands. The average Sb amounts for the 1, 2, 3, and 4-h persistence intervals were 0.08, 0.06, 0.04, and 0.03 μg , respectively; therefore, Sb appears useful while Pb does not. Our results are in agreement with the findings of Kilty [2], which also showed a drop in Sb of about an order of magnitude from the 0 to the 1-h samples. However, the residues produced by the 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.

An order-of-magnitude decrease of the average Pb amount in the firing samples from the amount found immediately after firing the .32 pistol (2.3 μg) would reduce the average Pb to below the average handblank level. This explains why the Pb levels seen at the 1-h interval are indistinguishable from the handblank level. On the nonfiring hand, Pb and Sb amounts in samples taken immediately after firing are indistinguishable from common handblank levels. Average Pb values at the 1, 2, 3, and 4-h intervals after shooting are indistinguishable from the common Pb handblank levels, and no individual Pb value from the nonfiring samples was greater than 0.95 μg . Although no Sb was detected in any of the four samples from nonfiring hands taken immediately after the one-round, one-hand .32 pistol firings, it was detected in two of five nonfiring samples taken for each of the 1, 2, and 3-h delay periods, demonstrating transfer of residue from firing to nonfiring hand by activity. Clearly, if the threshold value for positive Sb analysis is set at 0.1 or 0.2 μg , essentially all firing samples collected 1 h or more after firing will be judged negative.

Adhesive Residue Collection

The tape adhesive sample collection method is rapid and can be used in conjunction with various techniques of analysis such as inorganic luminescence, atomic absorption [4], chromatography, and scanning electron microscopy [5]. The examination of a sample with the scanning electron microscope (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 by 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 sampling techniques employed in gunshot residue collection are less convenient and not readily adaptable to SEM examination. Data obtained by the various collection techniques can be expected to differ quantitatively because nonidentical areas of the hand are sampled and the efficiency of retention for various components of the residue can be expected to vary from method to method.

Summary

More than 170 gunshot residue samples were analyzed by the inorganic luminescence technique to assess various factors affecting the utility of this and similar methods of elemental gunshot residue detection. Indoor firings consistently produced more residue in samples than did outdoor firings. Experiments were conducted to determine the effects of activity after firing on the ability to subsequently detect residue in samples. It was shown that residue can be transferred from hand to hand and can be detected in pockets under suitable conditions. Antimony, when present in the ammunition, was found to be the most useful indicator for the presence of residue, in that nearly all firings resulted in more Sb than was found in any of 45 handblank analyses obtained in these studies. Data obtained with a .32 pistol suggest that Sb normally exceeds environmental levels when residue is collected from the hand following delays up to several hours after firing. However, it will rarely exceed the currently used threshold levels of 0.1 to 0.2 μg . Therefore, elemental analysis is best suited to screening applications for which threshold levels can be greatly reduced.

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