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Analysis of Long-Range Bullet Entrance Holes by Atomic Absorption Spectrophotometry and Scanning Electron Microscopy

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ABSTRACT: Bullet residue and primer particles were analyzed by scanning electron microscopy with energy dispersive analysis (SEM-EDA) and by flame and flameless atomic absorption spectrophotometry (AAS). The residue and particles were on cloth targets around entrance holes produced by bullets fired at distances of 10 to 200 m. Primer particles and their chemical constituents were almost always detected by SEM-EDA around the holes produced by rifles and pistols fired at long ranges, and in many cases the barium and antimony associated with primer particles were detected by flameless AAS. Particles were also detected by SEM-EDA on the rear of bullets fired into and recovered from wooden blocks. Usually a hole caused by a bullet jacketed with gilding metal could be distinguished from one caused by a bullet jacketed with yellow brass alloy. Paint from bullet tips of military tracers was also detected. Analysis of the various residues around entrance holes provides a means for identifying the type of ammunition used.

KEYWORDS: criminalistics, ballistics, chemical analysis

This study deals with the analysis of gunshot residue around bullet entrance holes in cloth at firing distances of 10 to 200 m, using flame and flameless atomic absorption spectrophotometry (AAS) and scanning electron microscopy with energy dispersive analysis (SEM-EDA). Analysis of the residue can provide a basis for distinguishing among types of ammunition producing a bullet hole. This information can be particularly important in shooting incidents involving more than one type of firearm and ammunition, where the bullet has passed through the victim.

Although AAS [1, 2] and SEM-EDA [3-5] have been used to identify gunshot residues, very little information is available about their application to bullet entrance holes produced by rifles or pistols fired at long range. Measurement by AAS of the lead concentration in concentric rings around bullet entrance holes was used as a basis for determining firing distances up to 915 mm (36 in.); however, the circle containing the black ring with the bullet hole itself was discarded because the results were not reproducible [1]. Many of the applications of flameless AAS to gunshot residue analysis have concentrated on the determination of barium and antimony from specimens obtained from the hand holding the gun [2].

Analysis by SEM-EDA of microscopic bullet fragments recovered from the wound track of victims and the clothing from the exit wound has been reported [3]. Fragments composed of 95% copper and 5% zinc, 90% copper and 10% zinc, or 70% copper and 30% zinc were distinguished. Additional elements detected in the bullet residue included nickel, lead, an-

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timony, iron, and manganese. One case in which SEM-EDA has been used to differentiate bullet types presumed to be responsible for a given bullet hole has been reported by Keeley [4]. Based on the detection of copper in fibers removed from the bullet hole area, it was possible to distinguish between a .32-caliber soft-lead bullet and a .38-caliber copper jacketed bullet.

Concentration patterns of copper and antimony have been detected around bullet holes at firing distances of more than 3 m (10 ft) using neutron activation analysis (NAA). Again, the cloth area containing the bullet hole itself did not give good reproducibility, presumably because of the wiping action of the bullet on the target material [6]. Antimony has been detected by NAA on bullet entrance holes in cotton cloth at firing distances of up to 21 m (70 ft). Surface densities varied from 0.177 to 0.316 $\mu\text{g}/\text{cm}^2$, and no significant differences were detected as a function of firing distance in the range of 1.5 to 21 m (5 to 70 ft) [7].

Distribution patterns of muzzle deposits have been analyzed by SEM-EDA at firing distances up to 915 mm (36 in.) by suspending sampling disks at various positions around the bullet trajectory. The gunshot residue collected within 0.3 m (1 ft) of the muzzle consisted of primer and bullet residue, from 0.3 to 0.9 m (1 to 3 ft) the residue was almost entirely bullet particles, and at a distance greater than 0.9 m (3 ft) only a few bullet particles were found [5]. An investigation by NAA of gunshot residue distribution patterns on surfaces below the bullet trajectory detected barium and antimony at distances of 12 m (39 ft) from the muzzle [8].

Experimental Procedure

Tests shots were fired at an outdoors military range using 35- by 35-cm pieces of white cotton cloth as target material. The cloth targets were suspended from a 40- by 40-cm wooden frame. The test shots were performed with military and civilian firearms including revolvers, semiautomatic pistols, submachine guns, and semiautomatic and fully automatic rifles of Western and Eastern bloc manufacture. Ammunition included copper alloy jacketed bullets, iron alloy jacketed bullets, nonjacketed bullets, nickel plate jacketed bullets, copper alloy coated bullets, and tracers. Primers included noncorrosive and corrosive types, some using mercury fulminate as an initiator. Primer cases contained a nickel plating or were composed of a copper zinc alloy. The cartridge cases were composed of brass, but several contained a nickel plating (see Table 1 and Fig. 1).

A Perkin Elmer Model 403 atomic absorption spectrophotometer with the HGA Model 500 graphite furnace was used. Circles of 8-mm radius were cut out around the bullet entrance hole and leached for 2 h with 2 mL of a 1:3 (v/v) nitric acid solution, using analytical reagent-grade nitric acid. Background specimens, obtained by cutting an 8-mm-radius circle from an area of target cloth where a bullet had not penetrated, were run simultaneously. The circles were then removed from the solution, and the residue was analyzed.

A Cambridge Scanning Co. Cam Scan III S scanning electron microscope with a backscatter detector [9] and an Elscint Proxan energy dispersive analyzer were used. The cloth bullet hole specimens were coated with carbon prior to analysis by using a Polaron SEM Model E 5000 coating unit.

Results and Discussion

Transfer Mechanisms

The residue deposited around an entrance hole by the bullet would normally consist of a mixture of the following elements: copper, zinc, lead, antimony, and nickel. Iron would also be expected to be found for those types of ammunition containing an iron jacket or an iron alloy core instead of the more common lead-antimony alloy core. The residue is deposited as the result of a single direct transfer, namely, transfer of the bullet elements onto the target as the result of direct contact with the target.

TABLE 1—Composition

Number	Ammunition	Cartridge Case ^a	Primer Case ^a	Bullet Jacket ^b
1	5.56 Western Cartridge Corporation 1976	70% Cu 30% Zn	70% Cu 30% Zn	90% Cu 10% Zn
2	5.56 Twin City 1973	70% Cu 30% Zn	70% Cu 30% Zn	90% Cu 10% Zn
3	5.56 tracer, Twin City	70% Cu 30% Zn	70% Cu 30% Zn	90% Cu 10% Zn
4	5.56 Lake City 1973	70% Cu 30% Zn	70% Cu 30% Zn	90% Cu 10% Zn
5	5.56 tracer, Lake City 1975	70% Cu 30% Zn	70% Cu 30% Zn	Fe alloy
6	0.22 Remington high speed	70% Cu 30% Zn
7	0.22 Winchester X	70% Cu 30% Zn
8	0.22 Remington standard	70% Cu 30% Zn
9	7.62 × 39 Russian	90% Cu 10% Zn coating on Fe alloy	70% Cu 30% Zn	Fe alloy
10	7.62 Egyptian armor-piercing incendiary	70% Cu 30% Zn coating on Fe alloy	70% Cu 30% Zn	90% Cu 10% Zn
11	7.62 NATO FNM 74-50 (Portugese)	70% Cu 30% Zn	70% Cu 30% Zn	Fe alloy
12	0.30 carbine Western Winchester	70% Cu 30% Zn	Ni coating	90% Cu 10% Zn
13	7.65 Gevelot	70% Cu 30% Zn	70% Cu 30% Zn	70% Cu 30% Zn
14	7.65 Sellier and Bellot	70% Cu 30% Zn	70% Cu 30% Zn	Fe alloy
15	9-mm short Hirtenburg	70% Cu 30% Zn	Ni coated	Fe alloy
16	9-mm parabellum, Israeli manufacture 1975	70% Cu 30% Zn	70% Cu 30% Zn	90% Cu 10% Zn
17	9-mm parabellum tracer, Israeli manufacture	70% Cu 30% Zn	70% Cu 30% Zn	90% Zn 10% Zn
18	9-mm parabellum, Israeli manufacture 1978	70% Cu 30% Zn	Ni coated	90% Cu 10% Zn
19	9-mm parabellum, FN 1970	70% Cu 30% Zn	70% Cu 30% Zn	70% Cu 30% Zn
20	9-mm parabellum, Fiocchi 1950	70% Cu 30% Zn	70% Cu 30% Zn	Fe alloy
21	9-mm parabellum, Thorpe Arch 1957	70% Cu 30% Zn	Ni coated	90% Cu 10% Zn
22	0.38 Smith & Wesson Remington Peters	Ni coating on 70% Cu 30% Zn	Ni coated	...
23	0.38 Smith & Wesson Browning	70% Cu 30% Zn	Ni coated	...
24	0.38 Special, Peters	70% Cu 30% Zn	Ni coated	...
25	0.38 Special, homemade	Ni coating on 70% Cu 30% Zn	90% Cu 10% Zn	...
26	0.45 Western Cartridge Corp. 1964	70% Cu 30% Zn	70% Cu 30% Zn	90% Cu 10% Zn

^aYellow cartridge brass composition assumed to be 70% Cu 30% Zn [16].

^bResults based on analysis by AAS.

^cResults based on analysis by SEM-EDA.

In some cases an ablative transfer process is clearly involved. In test shots fired at 90° into cloth targets at ranges of 10 to 200 m, with fully jacketed bullets of a copper-zinc alloy, the lead and antimony from the bullet core are readily detected. Since the bullet core never comes in direct contact with the target cloth under these conditions, the most likely explanation for the presence of the lead and antimony is that continuous ablation of the bullet core takes place during its passage through the target, which results in the transfer of bullet core particles onto the target cloth. A similar transfer process seems to be the most probable explana-

of cartridges.

Bullet Coating ^b	Bullet Core ^b	Major Primer Elements ^c	Miscellaneous
...	Pb-Sb	Pb, Ba, Sb, Al	...
...	Pb-Sb	Pb, Ba, Sb, Al	...
...	Pb-Sb	Pb, Ba, Sb	red paint on bullet, Sr
...	Pb-Sb	Pb, Ba, Sb, Al	...
90%Cu 10%Zn	Pb-Sb	Pb, Ba, Sb	red paint on bullet, Sr
70%Cu 30%Zn	Pb-Sb	Pb	...
100%Cu	Pb-Sb	Pb, Ba	...
...	Pb-Sb	Pb	...
90%Cu 10%Zn	Pb-Sb layer on Fe alloy core	Hg, Cl, Sb, S, Sn	...
...	Fe alloy	K, Cl, Sb, S	red and black paint on bullet
90%Cu 10%Zn	Pb-Sb	Pb, Ba, Ca, Si	...
...	Pb-Sb	Pb, Ba, Sb, Al	...
Ni	Pb-Sb	Hg, K, Ba, Sb, S	...
90%Cu 10%Zn	Pb-Sb layer on Fe alloy core	Pb, Ba, Sn, Ca, Si	...
Ni and 90%Cu 10%Zn	Pb-Sb	Pb, Ba, Ca, Si	...
...	Pb-Sb	Pb, K, Cl, Sb, S	...
...	Pb-Sb	Pb, K, Cl, Sb, S	red paint on bullet, Sr
...	Pb-Sb	Pb, Ba, Sb, S, Al	silver paint on bullet
...	Pb-Sb	Pb, Ba, Sb	...
Ni	Pb-Sb	K, Cl, Sb, S	...
...	Pb-Sb	Hg, Pb, K, Cl, Sb, S	...
...	Pb-Sb	Pb, Ba, Sb, Al	...
...	Pb-Sb	Pb, Ba, Ca, Si	...
...	Pb-Sb	Pb, Ba, Sb	...
...	Pb-Sb	Pb, Ba, Ca, Si	...
...	Pb-Sb	Pb, Ba, Sb	...

tion for the detection of elements such as strontium from tracer bullets on cloth targets. Here a single-stage, but indirect, transfer process is involved.

Many of the elements mentioned above could also originate from sources other than the bullet. For example, iron present on a rusty gun barrel could be transferred onto the bullet during firing and then transferred to the cloth. This involves a double transfer. Even at long ranges the possibility should not be ruled out that copper and zinc found near entrance holes could have originated from the cartridge case. Lead could also originate from the lead styphnate in the primer mixture, and antimony could originate from the antimony sulfide found in many primers.

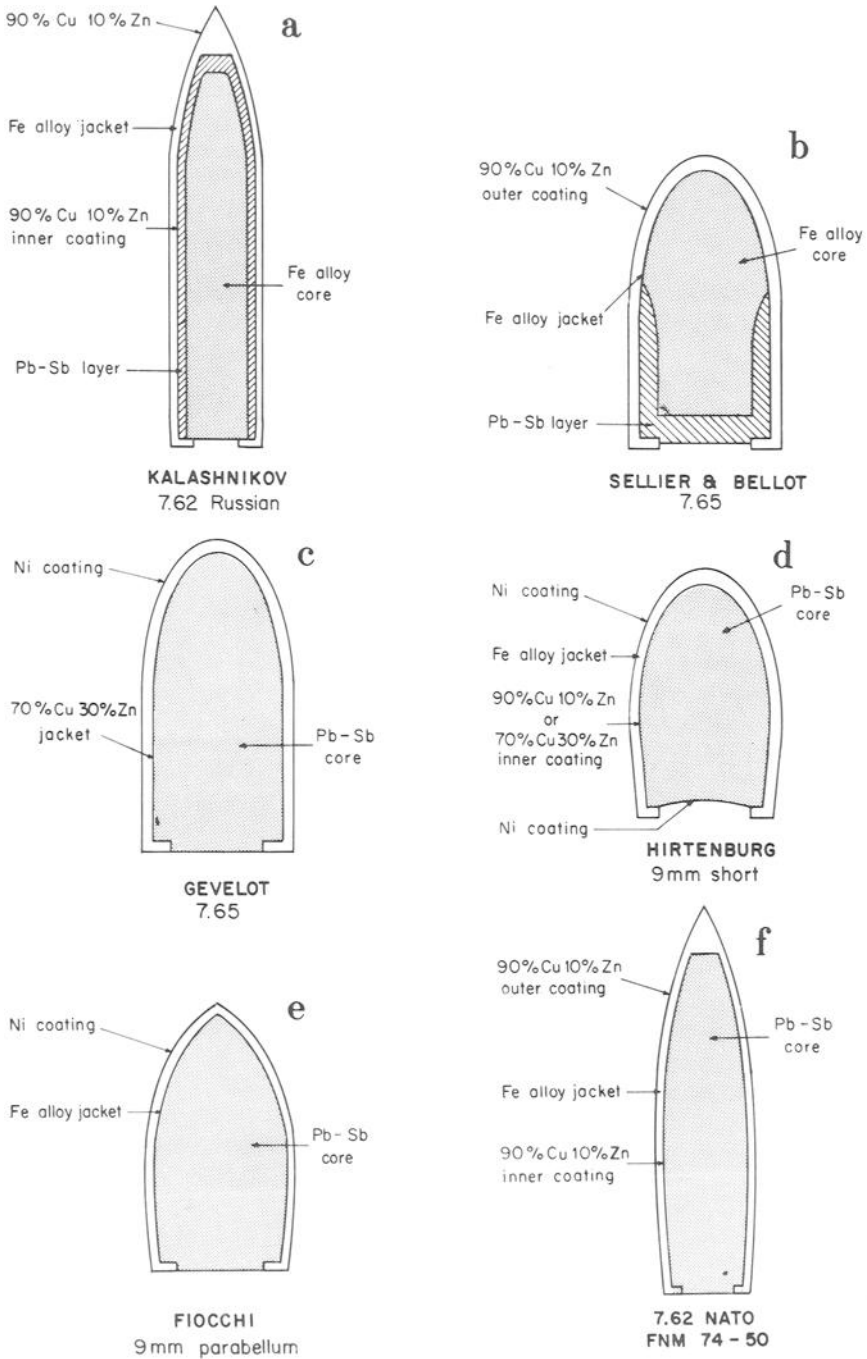


FIG. 1—Diagram indicating composition of six types of ammunition tested in the study.

Copper, Zinc, and Lead

In Table 2 the results of 30 test shots are given. The elemental concentrations are reported in parts per million in the test solution. Since 2 mL of solution were used and the surface area of an 8-mm-radius circle is 2.0 cm^2 , $\text{ppm} = \mu\text{g}/\text{cm}^2$. The copper and zinc concentrations are also reported as values normalized to 100%. Similarly, the lead plus antimony concentrations were normalized to 100%. Typical concentration ranges found in the blanks are reported at the end of the table.

The copper and zinc concentrations were normalized to 100% on the assumption that the major source of these two elements around a bullet entrance hole is the binary alloy. A total of 19 of the normalized copper-zinc values are close to 90% copper and 10% zinc, which is the composition of gilding metal, a standard alloy used in bullet jackets [10]. There are several notable exceptions. The .22-caliber Remington high-speed ammunition (test shots 6 and 7) gave normalized copper-zinc values close to 70% copper and 30% zinc. These bullets are coated with a yellow brass which appears to have the same color as the cartridge brass; thus, the data could be used to distinguish a hole caused by a Remington high-speed bullet from one caused by a Winchester X bullet, which has a coating of copper (see test shots 8 and 9). The 9-mm FN ammunition (test shot 23) also gave normalized copper-zinc values close to 70% copper and 30% zinc. Therefore these data could be used to differentiate a bullet hole produced by a 9-mm FN bullet from holes produced by many other types of 9-mm bullets with jacket compositions of gilding metal (see test shots 19, 20, 21, 22, 24, and 25). Test shots 16 and 17 also have normalized copper-zinc values close to 70% copper and 30% zinc, consistent with their jacket composition.

Some of the bullets in Table 2 consist only of a lead-antimony alloy (test shots 10, 11, 26, 27, 28, and 29), yet various amounts of copper and zinc were detected around the bullet entrance holes. One possible explanation is prior gun barrel contamination. The only other possible source for the copper and zinc around a bullet hole caused by .22-caliber rimfire Remington standard ammunition (test shots 10 and 11) is the cartridge case. In test shot 28, made with .38 Special Peters ammunition, the normalized copper-zinc values are very close to 70% copper and 30% zinc. The most probable source of this copper and zinc is the cartridge case. The primer case is an unlikely source, since it is nickel plated and the nickel concentration around the bullet entrance hole is very low. In test shot 29, with homemade .38 Special ammunition, the only likely source of the copper and zinc is the primer case or gun barrel since the cartridge case is coated with nickel. The amounts of copper and zinc detected in this test shot are almost negligible. In test shot 26, the source of the copper and zinc is apparently the gun barrel, since the cartridge case and primer case are nickel coated and an insignificant amount of nickel was detected. The source of the copper and zinc in test shot 27 is not clear.

There is very little chance that the data from the six test shots just described would be misinterpreted as indicating a copper-zinc alloy fully jacketed bullet for the following reasons:

1. The lead concentrations from these six test shots are high, varying from 16.0 to 57 ppm, as compared to ≤ 7.25 ppm for copper-zinc alloy jacketed bullets.
2. The lead/copper ratios are extremely high, ranging from 52:1 to 570:1 versus $\leq 2.4:1$ in test shots with copper-zinc alloy jacketed bullets.

Interpretation of the data is more difficult when results from soft-lead bullets coated with a copper-zinc alloy are compared; nevertheless, a basis for differentiation does exist. The lead concentration range of soft-lead bullets coated with a copper-zinc alloy is 5.7 to 17.6 ppm, and the lead/copper ratio ranges from 7:1 to 48:1 (see test shots 6, 7, 8, and 9 in Table 2 and Remington high-speed ammunition in Table 3). Using these criteria, it is possible to determine if an entrance hole was caused by .22 caliber Remington high speed, .22 caliber Winchester X, or Remington standard ammunition (test shots 6 to 11). The normalized copper-zinc results readily differentiate between the Remington high-speed and the Winchester X ammunition. The normalized copper-zinc results also readily differentiate between the Rem-

TABLE 2—Bullet entrance hole analysis by flameless AAS (corrected for background).

Number	Firearm, Ammunition, Range	Cu ppm ^d	Zn ppm ^d	Pb ppm ^b	Sb ppm ^b	Ni ppm	Fe ppm	Ba ppm	Sr ppm	K ppm	Sn ppm
1	M-16, 5.56 Western Cartridge Corp. 1976, 100 m	1.05 ^c 92.1%	0.09 ^c 7.9%	2.4 ^c 71.4%	0.96 28.6%	<0.014	~0.15 ^c	0.166	<0.01	ND ^d	<0.02
2	M-16, 5.56 Twin City 1973, 100 m	0.59 ^c 89.4%	0.07 ^c 10.6%	1.22 ^c 76.7%	0.37 23.3%	<0.025	<0.2 ^c	0.077	<0.02	ND	0.10
3	M-16, 5.56 tracer Twin City, 50 m	0.79 ^c 86.8%	0.12 ^c 13.2%	0.80 ^c >94.1%	<0.05 <5.9%	<0.025	<0.2 ^c	0.044	<0.05	ND	0.01
4	M-16, 5.56 Lake City 1973, 25 m	0.90 ^c 88.2%	0.12 ^c 11.8%	1.8 ^c 66.7%	0.90 33.3%	<0.03	ND	0.144	ND	ND	ND
5	M-16, 5.56 tracer Lake City 1978, 25 m	2.14 ^c 93.0%	0.15 ^c 7.0%	1.5 ^c 62.0%	0.92 38.0%	<0.03	~3.4	0.072	0.052	ND	<0.10
6	Ruger, 0.22 Remington high speed, 50 m	0.81 ^c 70.4%	0.34 ^c 29.6%	5.7 ^c 98.9%	0.064 1.1%	<0.03	8.4	<0.03	<0.03	ND	0.08
7	Ruger, 0.22 Remington high speed, 10 m	1.46 ^c 73.0%	0.54 ^c 27.0%	12.6 99.21%	0.10 0.79%	<0.010	1.4	0.031	<0.02	ND	0.06
8	Ruger, 0.22 Winchester X, 25 m	0.70 ^c 88.6%	0.09 ^c 11.4%	9.5 ^c >99.5%	<0.05 <0.5%	<0.025	<0.2 ^c	0.023	<0.05	0.31 ^{c,e}	0.10
9	Ruger, 0.22 Winchester X, 10 m	0.37 ^c 86.0%	0.06 ^c 14.0%	17.6 ^c 99.27%	0.13 0.73%	<0.03	3.5	0.125	0.02	ND	0.32
10	Beretta, 0.22 Remington standard, 25 m	0.15 62.5%	0.09 ^c 37.5%	~24 ^c 98.36%	0.40 1.64%	<0.016	<0.05	0.065	<0.01	0.16 ^{c,e}	0.03
11	Ruger, 0.22 Remington standard, 10 m	0.53 68.8%	0.24 ^c 31.2%	28.8 ^c 97.3%	0.80 2.7%	<0.03	1.4	0.144	<0.02	ND	0.13
12	Kalashnikov, 7.62 Russian, 100 m	5.46 ^c 88.9%	0.68 ^c 11.1%	0.15 ^c 2.8%	5.2 97.2%	<0.016	~0.62	<0.005	0.03	ND	≥4.6
13	Kalashnikov, 7.62 Egyptian armor piercing incendiary, 50 m	0.72 ^c 86.7%	0.11 ^c 13.3%	0.12 ^c 28.6%	0.30 71.4%	<0.014	~0.55	0.015	<0.01	0.90 ^{c,e}	0.38
14	FN rifle, 7.62 NATO FNM (Portugese), 100 m	6.20 ^c 88.2%	0.86 ^c 11.8%	4.95 ^c 97.44%	0.13 2.56%	<0.017	<0.2	0.090	0.003	ND	0.29
15	M-1 carbine, 0.30 Western Winchester, 100 m	3.91 ^c 87.7%	0.55 ^c 12.8%	2.8 ^c 81.4%	0.64 18.6%	0.03	4.9	0.338	0.03	ND	0.08
16	VZ-70, 7.65 Gevelot, 25 m	0.59 ^c 70.2%	0.25 ^c 29.8%	0.22 ^c 32.3%	0.42 67.7%	0.30	<0.05 ^c	0.065	ND	0.90 ^{c,e}	0.08 ^e

17	VZ-70, 7.65 Sellier and Bellot, 25 m	1.70 ^c 73.9%	0.60 26.1%	3.85 ^c 95.89%	0.17 4.11%	0.030	0.74	0.381	0.046	<0.6 ^{c,e,f}	1.41
18	Beretta, 9 mm short Hirtenburg, 25 m	0.55 ^c 72.4%	0.21 ^c 27.6%	1.18 97.5%	0.03 2.5%	0.238	0.5	0.166	<0.02	0.11 ^{c,e}	0.08
19	Uzi, 9-mm Israeli manufacture 1975, 50 m	7.27 ^c 90.1%	0.88 ^c 9.9%	1.40 ^c 55.8%	1.11 44.2%	<0.017	0.6	0.016	0.097	-0.42	0.17
20	Uzi, 9-mm tracer Israeli manufacture, 50 m	6.21 ^c 86.5%	0.97 ^c 13.5%	1.8 ^c 71.4%	0.72 28.6%	<0.025	0.9 ^c	<0.002	1.85 ^c	ND	0.01
21	Browning, 9-mm Israeli manufacture 1978, 25 m	5.24 ^c 88.2%	0.70 ^c 11.8%	0.8 ^c 69.0%	0.36 31.0%	<0.03	-0.6	0.02	0.012	0.15 ^{c,e,f}	0.04 ^e
22	Browning, 9-mm Israeli manufacture 1978, 25 m	7.84 ^c 88.5%	1.02 ^c 11.5%	2.4 89.9%	0.27 10.1%	<0.03	-0.2	0.94	<0.01	<0.7 ^{c,e,f}	0.14
23	Browning, 9-mm FN 1970, 25 m	2.55 ^c 70.6%	1.06 ^c 29.4%	1.85 84.9%	0.33 15.1%	<0.010	0.41	0.418	<0.02	0.2 ^{c,e,f}	0.07
24	Browning, 9-mm Fiocchi 1956, 25 m	4.99 ^c 92.8%	0.39 ^c 7.2%	0.55 ^c 26.0%	1.57 74.0%	0.84	≥5.0	0.11	<0.01	ND	0.04
25	Browning, 9-mm Thorpe Arch 1957, 25 m	4.26 ^c 86.9%	~0.64 ^c 13.1%	2.4 ^c 64.3%	1.33 35.7%	0.045	<0.015	0.10	<0.01	0.90 ^{c,e,f}	0.10
26	Webley, 0.38 Smith & Wesson Remington Peters, 25 m	0.10 ^c 71.4%	0.04 ^c 28.6%	57 96.35%	2.16 3.65%	<0.010	2.6	0.756	<0.02	1.9 ^{c,e}	0.08
27	Webley, 0.38 Smith & Wesson, Browning, 25 m	0.68 86.5%	0.106 ^c 13.5%	56.4 ^c 96.05%	2.32 3.95%	<0.015	0.11	0.200	0.007	≤0.15	0.41
28	Colt, 0.38 Special Peters, 25 m	0.69 ^c 67%	0.34 ^c 33%	~36 93.0%	2.67 7.0%	<0.016	<0.05 ^c	1.12	<0.01	ND	0.05
29	Colt, 0.38 Special homemade, 25 m	0.03 75%	0.01 ^c 25%	16.0 ^c 96.0%	0.66 4.0%	<0.017	-0.6	0.120	0.001	ND	0.02
30	Colt, 0.45 Western Cartridge Corp. 1964, 25 m	3.00 ^c 89.8%	0.34 ^c 10.2%	7.25 ^c 65.8%	3.76 34.2%	<0.017	-0.2	0.98	0.013	1.0 ^{c,e,f}	<0.10
31	Range of cloth blanks	<0.02 ^c -0.24 ^c	0.071 ^c -0.26	<0.05 ^c -0.21 ^c	<0.01 -0.12	<0.05	-0.15 ^c -4.1	0.012 -0.075	0.04 -0.32	0.49 ^c -9.7 ^c	0.04 -0.13

^aThe data on the second line of each test shot are the normalized Cu-Zn values.

^bThe data on the second line of each test shot are the normalized Pb-Sb values.

^cFlame AAS.

^dND = not determined.

^eAnalysis performed on separate test shot.

^fHigh background.

TABLE 3—*Reproducibility of results of bullet entrance*

Number	Firearms, Ammunition, Range	Cu ppm ^a	Zn ppm ^a	Pb ppm ^b
1	VZ-70, 7.65 Gevelot, 25 m	0.59 ^c	0.29 ^c	0.32 ^c
		67.0%	33.0%	32.0%
2	VZ-70, 7.65 Gevelot, 25 m	0.67 ^c	0.39 ^c	0.27 ^c
		63.2%	36.8%	45.8%
3	VZ-70, 7.65 Gevelot, 25 m	0.70 ^c	0.28 ^c	0.20 ^c
		71.4%	28.6%	16.9%
4	VZ-70, 7.65 Gevelot, 25 m	1.04 ^c	0.50 ^c	0.32 ^c
		67.5%	32.5%	34.0%
5	VZ-70, 7.65 Gevelot, 25 m	0.59 ^c	0.25 ^c	0.20 ^c
		70.2%	29.8%	29.0%
...	Average value $\pm \sigma^e$	0.72 \pm 0.19	0.34 \pm 0.10	0.27 \pm 0.05
		67.9 \pm 3.2%	32.1 \pm 3.2%	31.5 \pm 10.4%
1	M-1 Carbine, 0.30 Western Winchester, 100 m	3.97 ^c	0.48	1.85 ^c
		89.2%	10.8%	77.1%
2	M-1 Carbine, 0.30 Western Winchester, 100 m	5.1	0.53	7.15 ^c
		89.6%	10.4%	92.2%
3	M-1 Carbine, 0.30 Western Winchester, 100 m	5.37 ^c	0.58 ^c	3.8 ^c
		90.3%	9.7%	69.1%
4	M-1 Carbine, 0.30 Western Winchester, 100 m	3.93 ^c	0.55 ^c	2.75 ^c
		87.7%	12.3%	81.1%
5	M-1 Carbine, 0.30 Western Winchester, 100 m	4.47 ^c	0.49 ^c	2.8
		90.1%	9.9%	63.5%
...	Average value $\pm \sigma$	4.57 \pm 0.65	0.53 \pm 0.04	3.7 \pm 2.1
		89.4 \pm 1.0%	10.6 \pm 1.0%	76.6 \pm 11.1%
1	Ruger, 0.22 Remington high speed, 50 m	0.81 ^c	0.34 ^c	5.7 ^c
		70.4%	29.6%	98.88%
2	Ruger, 0.22 Remington high speed, 50 m	0.88 ^c	0.38	9.5 ^c
		69.8%	30.2%	99.28%
3	Ruger, 0.22 Remington high speed, 50 m	1.01 ^c	0.46 ^c	10.9 ^c
		68.7%	31.3%	98.7%
4	Ruger, 0.22 Remington high speed, 50 m	0.87 ^c	0.58	6.2 ^c
		60.0%	40.0%	99.11%
5	Ruger, 0.22 Remington high speed, 50 m	1.19 ^c	0.50 ^c	10.2
		70.4%	29.6%	99.13%
...	Average value $\pm \sigma$	0.95 \pm 0.15	0.45 \pm 0.10	8.5 \pm 2.4
		67.9 \pm 4.4%	32.1 \pm 4.4%	99.0 \pm 0.23%

^aThe data on the second line of each test shot are the normalized Cu-Zn values.

^bThe data on the second line of each test shot are the normalized Pb-Sb values.

^cFlame AAS.

^dND = not determined.

^e σ = standard deviation.

ington standard and the Winchester X. What primarily differentiates the Remington standard from the Remington high-speed ammunition is the much higher lead concentration found around the bullet entrance hole from the Remington standard ammunition (24 to 28.8 ppm versus 5.7 to 12.6 ppm in Remington high-speed ammunition) and the much higher range for the lead/copper ratio found around the bullet entrance hole from Remington standard ammunition (54:1 to 160:1 versus 7.0:1 to 10.8:1 in Remington high-speed ammunition).

Antimony and Lead

In many cases excessively large amounts of antimony, when normalized to 100% with the lead concentration, were found around the bullet entrance hole. Since most bullet lead contains up to 5% antimony [11-13], it seems unreasonable to assume that the sole source of the antimony is bullet lead, especially in those cases where the normalized antimony value is 10% or more. In 7.62 \times 39 Russian ammunition, which contains an iron alloy core, the normalized antimony values are 97.2 and 71.4% for test shots 12 and 13, respectively. A total of 16 of the 30 test shots in Table 2 have normalized antimony values of 10% or above.

hole analysis by flameless AAS (corrected for background).

Sb ppm ^b	Ni ppm	Fe ppm	Ba ppm	Sr ppm	Sn ppm
0.68 68.0%	0.25	~0.05 ^c	0.098	<0.015	0.11
0.32 54.2%	0.57	~0.12 ^c	0.088	0.027	0.12
0.98 83.1%	0.30	0.09	0.156	<0.02	0.11
0.62 66.0%	0.89	~0.37 ^c	0.034	<0.015	0.10
0.49 71.0%	0.31	~0.05 ^c	0.068	ND ^d	ND
0.62 ± 0.24 68.5 ± 10.4%	0.46 ± 0.27	0.14 ± 0.13	0.089 ± 0.045	0.02 ± 0.006	0.11 ± 0.01
0.55 22.9%	<0.03	0.2	0.087	<0.03	0.03
0.60 7.8%	0.08	4.2	0.118	0.03	0.07
1.70 30.9%	<0.010	0.4	0.166	<0.04	ND
0.64 18.9%	0.03	4.9	0.338	0.03	0.08
1.61 36.5%	<0.010	0.3	0.145	<0.02	0.06
1.02 ± 0.58 23.4 ± 11.1%	0.03 ± 0.03	2.0 ± 2.4	0.171 ± 0.098	0.03 ± 0.007	0.06 ± 0.02
0.064 1.12%	<0.03	5.4	<0.03	<0.03	0.08
0.068 0.72%	<0.03	5.4	0.051	0.04	0.09
0.142 1.3%	<0.03	4.3	<0.03	<0.03	ND
0.055 0.89%	0.03	1.7	0.086	<0.03	<0.03
0.09 0.87%	<0.010	4.2	0.016	<0.02	0.03
0.084 ± 0.035 0.98 ± 0.23%	0.03 ± 0.01	4.2 ± 1.5	0.043 ± 0.027	0.03 ± 0.01	0.06 ± 0.03

One explanation for these high values is that the antimony sulfide in the primer mixture acts as a source of antimony. Primer particles obviously do not travel 10 to 200 m as a result of the pressure built up in the gun barrel after the initial ignition and while the bullet is accelerating in the gun barrel. The generally accepted maximum distance that such primer particles might travel is on the order of 5 m [6], much less than the ranges used in these test firings. However, during the time that the bullet is accelerating in the gun barrel, the various inorganic compounds and organic salts present in the primer or the gunpowder decompose into a variety of products. Some of these decomposition products are no doubt the metallic element itself and various forms of its oxides. Recombinations of the metallic elements and their oxides in the vapor phase are also possible [14, 15]. At the same time, the accelerating bullet is under very high pressure and temperature, and the lead-antimony core is in a soft if not near-molten state. In the time spent by the bullet in the barrel, there may be a physical interaction between the metallic primer decomposition products and the rear of the bullet, resulting in the adhesion of numerous primer decomposition products to the rear of the bullet. During the flight of the bullet, continuous ablation occurs, resulting in a transfer of these primer particles to the target cloth.

In an attempt to verify this hypothesis, a test shot was fired a distance of 25 m into a block of wood. An Uzi submachine gun and fully jacketed 9-mm noncorrosive Israeli ammunition (see Table 1, number 18) were used. The bullet was recovered from the wood, and the area of the base of the bullet was carefully examined under the scanning electron microscope. Several spherical particles having the elemental composition characteristic of the primer were ob-

served (see Fig. 2). A similar experiment was performed at a distance of 15 m with an Enfield revolver and .38-caliber Remington Peters ammunition (see Table 1, number 22). Smears of lead and barium were readily observed on the rear of the bullet. The only possible source of barium was the primer (see Fig. 3). The results tend to confirm the hypothesis.

Nickel, Iron, and Strontium

Three of the bullets in Table 2 are nickel plated (test shots 16, 18, and 24). The nickel concentration around the bullet holes from these test shots ranged from 0.24 to 0.84 ppm. In test shots where the primer case, cartridge case, or primer and cartridge case were coated with nickel, no significant amounts of nickel were detected (test shots 15, 22, 25, 26, 27, 28, and 29). No test shots for which the nickel concentration was determined to be >0.05 ppm were false positives, thus providing a clear basis for distinguishing if a bullet hole was caused by a nickel-plated bullet or not. Inclusion of additional data from Table 3 results in only one false positive, if 0.05 ppm is chosen as the lower limit for significant results. Nickel is also readily detected by SEM-EDA, associated with the other elements found in the bullet and some of the elements found in the primer (see Fig. 4).

Two of the cartridges in Table 2 contain an iron core, jacket, and cartridge case (test shots 12 and 13), one of the bullets contains an iron core and jacket (test shot 17), and four of the bullets contain iron jackets (test shots 5, 14, 18, and 24). There appears to be very little interpretive value to the iron determination, since iron concentrations equal to or greater than those found for these seven test shots were measured in several other test shots, probably from rust from the gun barrel.

Three of the bullets in Table 2 are tracers (test shots 3, 5, and 20). The Uzi tracer (test shot 20) produces a very high strontium concentration around the bullet's entrance hole. The



FIG. 2—A 9-mm Israeli bullet fired by Uzi submachine gun at a range of 25 m. Photograph shows spherical primer particles on bullet's core. Spectrum is for the middle particle.

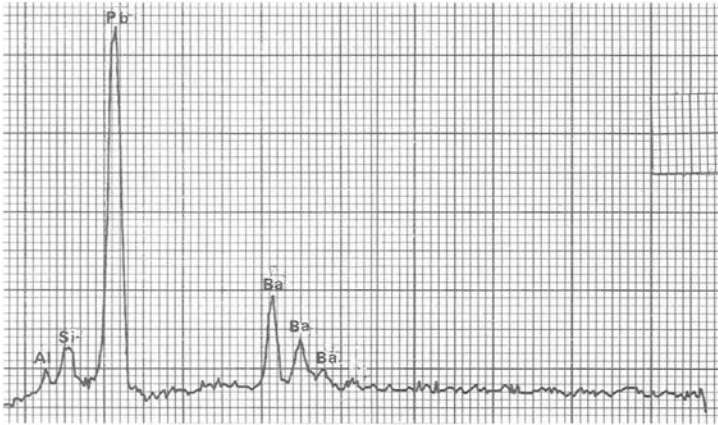


FIG. 3—Spectrum for .38-caliber Remington Peters ammunition fired by Enfield revolver at range of 15 m. Smear on rear of bullet was analyzed.



FIG. 4—Spectrum for Hirtenburg 9-mm short ammunition fired by Beretta pistol at range of 25 m.

results are far less clear for the two 5.56-caliber tracers. Examination with the scanning electron microscope of the two 5.56 tracer bullet holes reveals the presence of strontium in extremely low concentrations and associated with magnesium and chloride (see Fig. 5).

Paint on Bullet Tips

The tips of most military tracers are painted to distinguish them from standard ammunition, for example, the two 5.56-mm cartridges tested in this study had red tips. Frequently, a paint smear can be observed around the entrance hole caused by a tracer, providing an additional parameter for identifying the type of bullet which caused a given bullet hole. Analysis by SEM-EDA showed that the inorganic compositions of the red paint smears produced by the two tracers are not the same (see Figs. 6 and 7). The results of the paint analysis for one tracer showed that the paint in the smear around the bullet hole was the same paint scraped off of the bullet (see Figs. 7 and 8), indicating that no detectable change occurred in the paint composition as a result of the firing process.

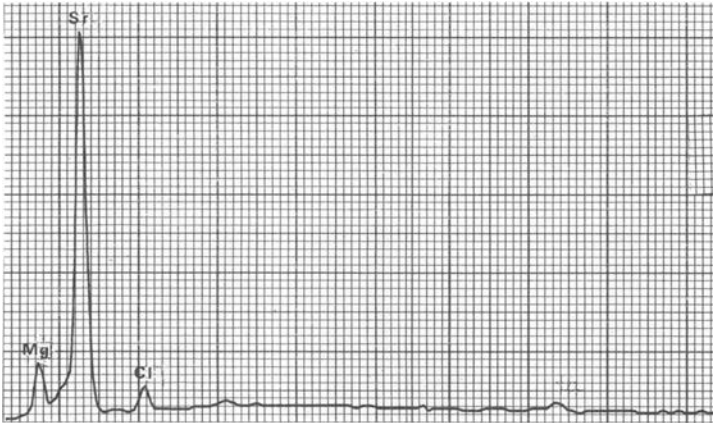


FIG. 5—Spectrum for 5.56 Twin City tracer fired by M-16 rifle at range of 50 m.

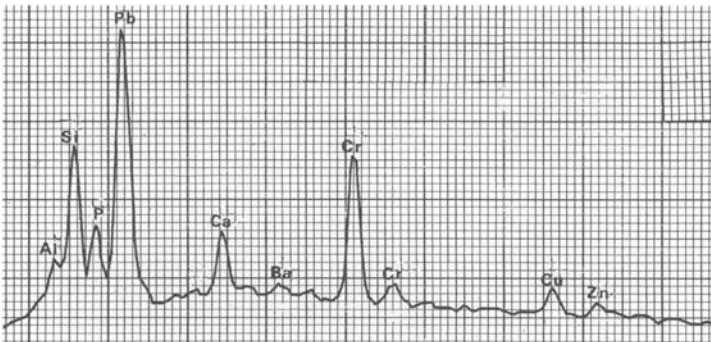


FIG. 6—Spectrum for 5.56 Lake City tracer (red paint around bullet hole) fired by M-16 rifle at range of 25 m.

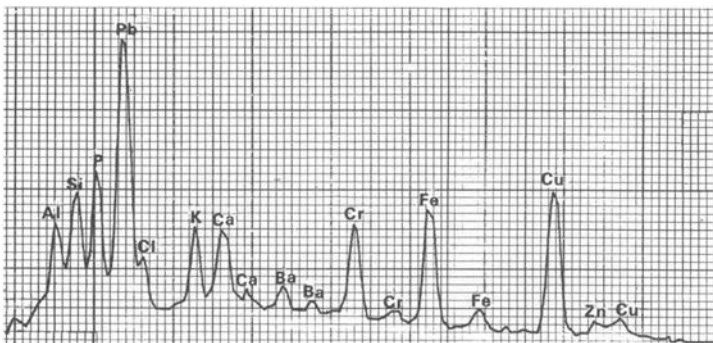


FIG. 7—Spectrum for 5.56 Twin City tracer (red paint around bullet hole) fired by M-16 rifle at range of 50 m.

Other special types of military ammunition are also frequently painted various colors. For example, the tip of the bullet of a 7.62 × 39-mm Egyptian armor-piercing incendiary round for the Kalashnikov rifle is painted red and black. Analysis of the paint by SEM-EDA revealed silicon and organic matter. This is of very limited value for identification purposes, except to distinguish it from another specimen.

Tin, Barium, and Potassium

Tin was detected in two of the shots listed in Table 2 (test shots 12 and 17). This element is rare in most types of Western ammunition but is still common in ammunition manufactured in Russia or Eastern bloc countries. The tin concentration is significantly high in test shots 12 and 17, and its presence is also readily detected by SEM-EDA (see Fig. 9).

The interpretation of the barium results is problematic. In the concentration range of 0.05 to 0.10 ppm, the results are inconclusive. Six of the 30 test shots in Table 2 fall within this range. At concentrations <0.05 ppm, two of the nine test shots gave false negatives (test shots 3 and 8). The results of analysis by SEM-EDA confirm that barium is indeed present in these two test shots (see Figs. 10 and 11). At concentrations >0.10 ppm, two of the 15 test shots gave false positives (test shots 11 and 24). This was confirmed by the failure to detect by SEM-EDA barium particles associated with other elements characteristic of gunshot residue. Detection of barium by flameless AAS alone should be considered as a preliminary positive result; corroboration by SEM-EDA analysis increases the reliability of the finding. Additional

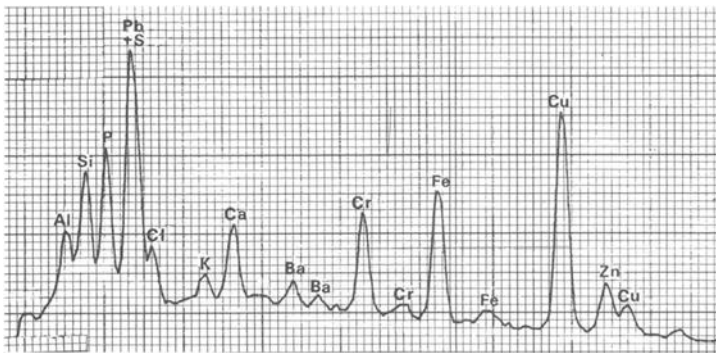


FIG. 8—Spectrum for paint from tip of 5.56 Twin City tracer.

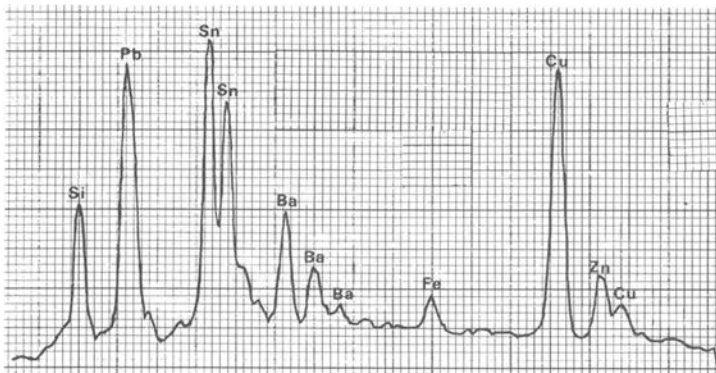


FIG. 9—Spectrum for Sellier and Bellot 7.65 ammunition fired by VZ 70 pistol at range of 25 m.

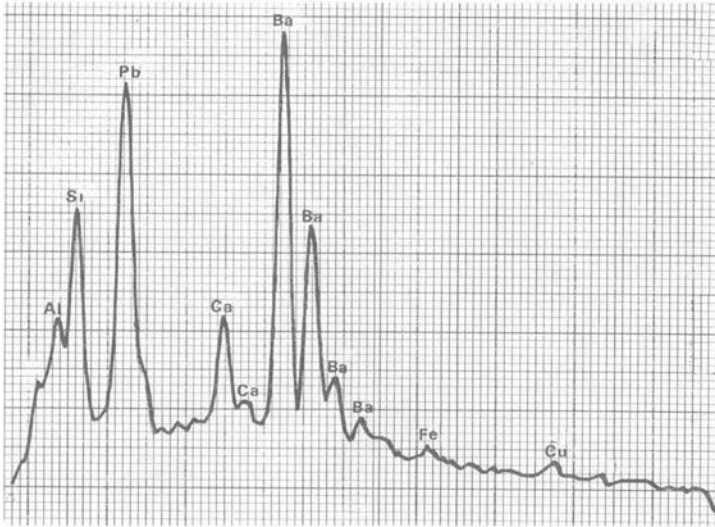


FIG. 10—Spectrum for .22-caliber Winchester X ammunition fired by Ruger pistol at range of 50 m.

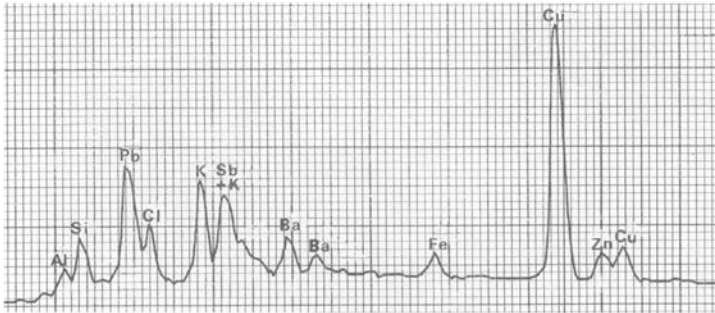


FIG. 11—Spectrum for 5.56 Twin City tracer fired by M-16 rifle at range of 50 m.

examples of barium in the gunshot residue around a bullet hole are illustrated in Figs. 12 and 13, including the spectra for a bullet fired from an M-16 at a distance of 200 m.

Fourteen of the test shots listed in Table 2 were analyzed for potassium. Five are known to contain this element in the primer (test shots 13, 16, 19, 21, and 25). Numerous contamination problems were encountered with reagents, equipment, and background when analyzing the solutions by flameless AAS. In an attempt to overcome many of these problems, separate test shots were performed, and the potassium density was determined by regular AAS. Severe background problems still remained, with six test shots having background levels ≥ 7 ppm and eight test shots having background levels ≤ 1 ppm. Of the eight test shots with low background levels, three contained potassium. If levels < 0.35 ppm are considered insignificant, one false positive and no false negatives were obtained. Potassium determinations are generally more reliable when SEM analysis is used (see Figs. 14 and 15).

Reproducibility of Results

To determine the reproducibility of the flameless AAS technique, a series of five test shots was performed with each of the following three types of ammunition: a copper alloy fully

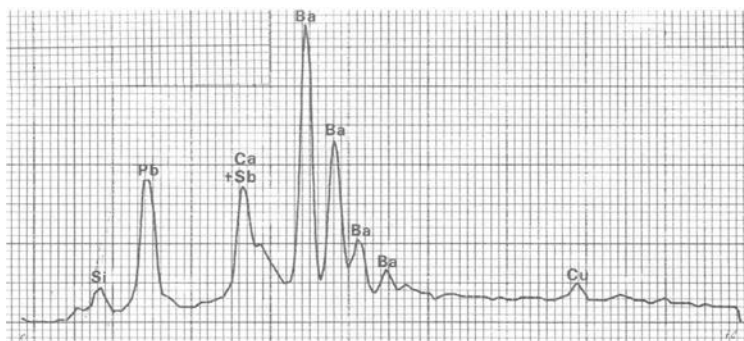


FIG. 12—Spectrum for .38 Special Peters ammunition fired by Colt revolver at range of 25 m.

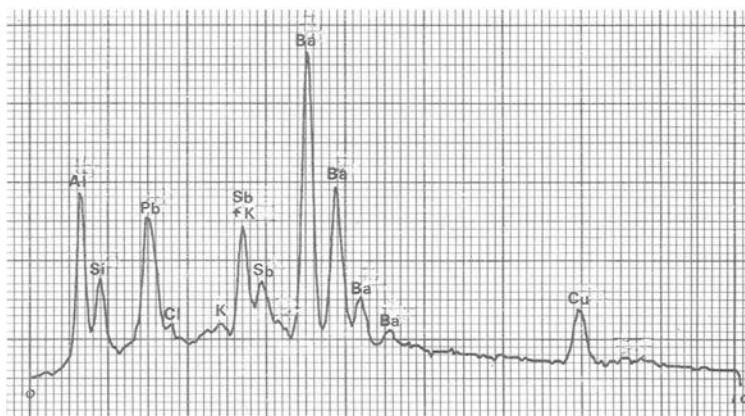


FIG. 13—Spectrum for 5.56 Western Cartridge Corp. ammunition fired by M-16 rifle at range of 200 m.



FIG. 14—Spectrum for 7.62 armor-piercing incendiary ammunition (Egyptian) fired by Kalishnikov rifle at range of 50 m.

TABLE 4—Bullet entrance hole analysis by flameless AAS:

Number	Firearm, Ammunition, Range	Cu ppm ^a	Zn ppm ^a	Pb ppm ^b
1	Uzi, 9-mm Israeli manufacture	7.27 ^c	0.80 ^c	1.40 ^c
	1975, 50 m, 90°	90.1%	9.9%	55.8%
2	Uzi, 9-mm Israeli manufacture	6.52 ^c	0.71 ^c	1.40 ^c
	1975, 50 m, 90°	90.2%	9.8%	59.6%
3	Uzi, 9-mm Israeli manufacture	12.0 ^c	1.30 ^c	2.10 ^c
	1975, 50 m, 90°	90.7%	9.3%	62.7%
4	Uzi, 9-mm Israeli manufacture	5.29 ^c	0.58 ^c	1.20 ^c
	1975, 50 m, 90°	90.1%	9.9%	64.2%
5	Uzi, 9-mm Israeli manufacture	8.06 ^c	0.88 ^c	1.30 ^c
	1975, 50 m, 90°	90.2%	9.8%	60.2%
...	Average ± σ ^d	7.82 ± 2.53	0.85 ± 0.24	1.48 ± 0.36
		90.3 ± 0.25%	9.7 ± 0.25%	60.7 ± 3.2%
1	Uzi, 9-mm Israeli manufacture	9.34 ^c	1.03 ^c	1.55 ^c
	1975, 50 m, 45°	90.1%	9.9%	57.6%
2	Uzi, 9-mm Israeli manufacture	6.48 ^c	0.81 ^c	1.48 ^c
	1975, 50 m, 45°	88.9%	11.1%	72.5%
3	Uzi, 9-mm Israeli manufacture	6.63 ^c	0.74 ^c	1.80 ^c
	1975, 50 m, 45°	90.0%	10.0%	66.9%
4	Uzi, 9-mm Israeli manufacture	5.97 ^c	0.63 ^c	1.33 ^c
	1975, 50 m, 45°	90.5%	9.5%	65.5%
5	Uzi, 9-mm Israeli manufacture	9.14 ^c	0.93 ^c	1.50 ^c
	1975, 50 m, 45°	90.8%	9.2%	62.5%
...	Average ± σ	7.5 ± 1.60	0.83 ± 0.16	1.58 ± 0.17
		90.1 ± 0.72%	9.9 ± 0.72%	65.0 ± 5.5%

^aThe data on the second line of each test shot are the normalized Cu-Zn values.

^bThe data on the second line of each test shot are the normalized Pb-Sb values.

^cFlame AAS.

^dσ = standard deviation.



FIG. 15—Spectrum for 9-mm Israeli ammunition fired by Uzi submachine gun at range of 50 m.

45° versus 90° entrance angle (corrected for background).

Sb ppm ^b	Ni ppm	Fe ppm	Ba ppm	Sr ppm	Sn ppm
1.11 44.2%	≤0.017	0.58	0.016	0.097	0.15
0.95 40.4%	≤0.017	0.28	0.060	0.003	0.18
1.25 37.3%	≤0.017	1.38	0.077	0.025	0.19
0.67 35.8%	≤0.010	0.28	<0.016	<0.02	0.06
0.84 39.8%	≤0.017	0.94	0.034	0.034	0.02
0.96 ± 0.23 39.3 ± 3.2%	≤0.017	0.68 ± 0.46	0.04 ± 0.027	0.036 ± 0.036	0.12 ± 0.08
1.14 42.4%	≤0.017	0.23	0.028	0.009	<0.01
0.56 27.5%	≤0.017	1.29	0.014	<0.001	0.01
0.89 33.1%	≤0.017	<0.3	0.014	0.005	0.01
0.70 34.5%	≤0.017	0.28	0.008	0.005	0.04
0.90 37.5%	≤0.017	1.99	0.014	0.009	<0.01
0.84 ± 0.22 35.0 ± 5.5%	≤0.017	0.82 ± 0.79	0.016 ± 0.008	0.006 ± 0.003	0.016 ± 0.013

jacketed bullet (.30-caliber carbine Western Winchester), a nickel-plated fully jacketed bullet (7.65 Gevelot), and a soft-lead bullet with a copper alloy coating (.22-caliber Remington high speed). The results are summarized in Table 3.

The advantages of normalizing the copper-zinc concentrations become readily obvious. Although on a given type of ammunition the range between the minimum and maximum copper concentrations may approach a factor of two (Gevelot ammunition), the standard deviation on the normalized data is ±3.2% absolute. The average normalized lead-antimony concentrations clearly show that the 7.65 Gevelot and .30-caliber carbine Western Winchester ammunition contain antimony in the primer (antimony >10%), even though some of the individual test shots have antimony values <10%. A normalized antimony value <10% does not necessarily mean that antimony is not present in the primer, especially if the absolute lead value is very high. However, the converse is generally true, that is, an antimony value >10% is generally a very strong indication that the ammunition involved does contain antimony in the primer. In the case of .22-caliber Remington high-speed ammunition, the normalized antimony value is very close to the reported level of antimony (0.91%) in the bullet lead [8].

From the nickel results, it is clear that the 7.65 Gevelot ammunition contains nickel and the other two types of ammunition do not. There does not appear to be any particular interpretive value to the iron determinations. The barium results indicate that the .30-caliber carbine Western Winchester ammunition does contain barium, the Remington high-speed ammunition does not, and the results on the Gevelot ammunition are inconclusive. However, this should be considered as a preliminary result to be verified by SEM-EDA. The strontium and tin values show the range in results expected in ammunition not containing these two elements. No significant difference was found in elemental concentrations by varying the angle of entry from 90° to 45° (see Table 4).

Additional Information from SEM-EDA Spectra

Analysis by SEM-EDA of gunshot residues around entrance holes made by bullets fired at long range can complement the data obtained by flameless AAS. The spectra frequently provide additional useful information about the ammunition composition, which would be difficult to deduce from flameless AAS, partially because of the high probability of serious background contamination. For example, calcium silicide is frequently found in primers, as is shown clearly by test shot 14 in Table 2 (see Fig. 16). Sometimes additives such as aluminum (see Fig. 13) or strontium associated with magnesium and chloride in 5.56-mm caliber tracer ammunition can be readily detected.

One element found in some primers from Eastern bloc countries, old Western bloc ammunition, and Russian ammunition is mercury. This element proved to be extremely difficult to detect by SEM-EDA; it was not detected even after several hours of searching the area around a bullet hole produced by a cartridge known to contain mercury in the primer. This was the only element commonly associated with gunshot residues for which this problem occurred.

A measure of the degree of differentiation that can be obtained, utilizing the methods and principles discussed in this article, among types of ammunition is illustrated in Fig. 17. From a total of 30 test shots involving 26 different types of ammunition, 17 classification groups containing one or more test shots and 12 classification groups containing only one test shot were obtained.

Conclusions

Primer particles of various compositions can readily be detected by SEM-EDA around entrance holes produced in cotton cloth by bullets from various guns fired at ranges of up to 200 m. Primer particles can also be detected by SEM-EDA on the base of the recovered bullet. Elements associated with primer compounds have been detected around bullet entrance holes by flameless AAS. The various elements associated with the bullet jacket, coatings, and core can be detected by either or both methods. The only common primer element not detectable was mercury. Normalization of the data for elements commonly associated as alloys, for example, copper-zinc and lead-antimony, facilitates interpretation of the data from flameless AAS and provides additional useful information. Copper-lead ratios are also of value, even though the absolute reproducibility of each element may vary widely from shot to shot. Strontium in tracers and paint on bullet tips also provide additional parameters for differentiating between types of ammunition that have caused a given bullet hole. Shots fired at entrance angles of 45° and 90° produced no significant differences in results.

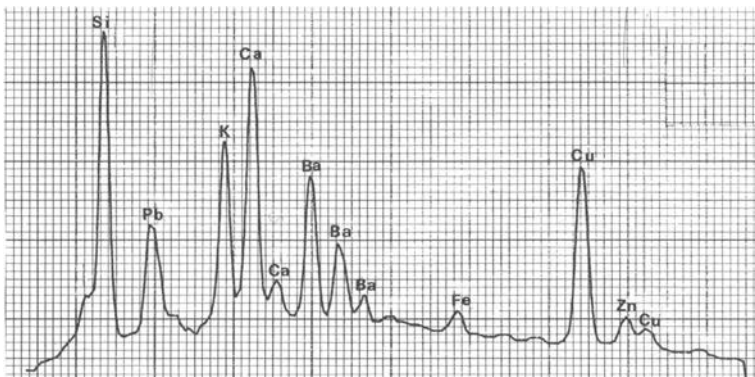


FIG. 16—Spectrum for 7.62 NATO (Portuguese) ammunition fired by FN rifle at range of 100 m.

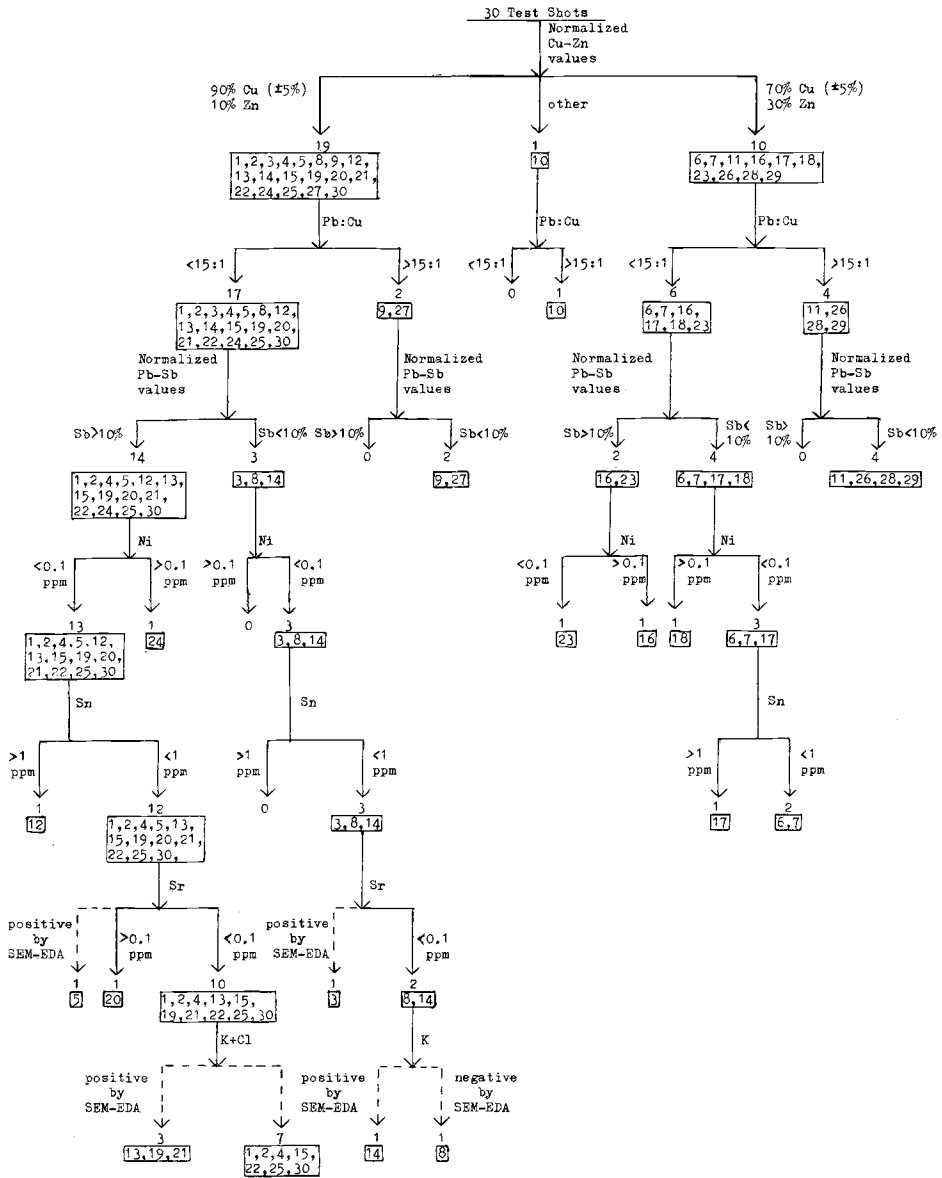


FIG. 17—Results of analyses by flameless AAS and SEM-EDA of residues from 30 test shots. The number above each block refers to the total number of shots included in each category; the numbers inside each block refer to the test shot number.

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