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TECHNICAL NOTE

CRIMINALISTICS

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Identification of Gunshot Residues in Fabric Targets Using Sector Field Inductively Coupled Plasma Mass Spectrometry Technique and Ternary Graphs*

ABSTRACT: During criminal investigations involving firearms, the detection of gunshot residues (GSRs) is one of the most important evidences. In the present study, a new method to identify trace evidences of GSRs, deposited around the bullet entrance hole, in different types of fabrics used as targets, is described. The experiments were carried out using a 0.38-inch caliber revolver, and 9-mm and 0.40-inch caliber pistols. Testimonies of 2.25 cm^2 of the fabrics were cut around the bullet entrance and digested with 10% nitric acid. Antimony, barium, and lead were analyzed in the remaining solution using a sector field inductively coupled plasma mass spectrometer. The concentrations of the elements were detected at levels up to few microgram per square centimeter. The use of ternary graphics allowed us to identify specific patterns of distribution for blank samples and the clear distinction between the revolver and pistols used.

KEYWORDS: forensic science, gunshot residues, sector field inductively coupled plasma mass spectrometry, fabric, firing arms, barium, lead, antimony, ternary graphs

When a weapon is fired, accompanying the projectile, vapors and particulate materials are expelled by the muzzle, barrel, and other openings present in the firearm. These particles, known as gunshot residues (GSRs), are composed of burnt and unburnt particles from the propellant, components from the primer, and contain elements from the projectile, cartridge case, plus previous residues present in the barrel. The GSRs may adhere to the skin or the clothes of the person who discharged the firearm, or on other people and objects in close proximity at the scene. Their precise identification constitutes important evidence in criminal investigation involving firearms (1,2). For this reason, during the last decades, several methodologies (3–5) have been used in forensic laboratories to determine the presence of GSRs on a wide variety of surfaces, for instance, shooter hands (6–12), firearms and ammunitions (11–13), vehicles and facilities (14), and around the bullet entrance hole in fabrics (15–18). For this purpose, several techniques have been used, based, mainly, on the identification of lead, barium, and antimony in the evidence under investigation (19–21). However, the above-mentioned elements may be easily found in the environment from other sources not related to the firearms discharge. This is especially troublesome when the use of atomic spectroscopy techniques, where the sample material undergoes a complete dissolution prior to the determination of the elements of interest, loses morphological information and individual particle identification (6,22–27). In the last years, emphasis has been placed on the use of scanning electron microscopy equipped with an energy-dispersive X-ray technique (SEM/EDS) for GSR analysis (11,28). This technique is nondestructive and provides both morphological and elemental profiles of individual particles. However, despite these advantages, the examination of a sample may take several hours, even using an automated system, and is strongly dependent on the quality and uniformity of the particulate on the adhesive surface used for sample collection (29,30).

This is especially worrying in a context of high levels of criminality and use of firearms. A recent document published by the United Nation Educational, Scientific and Cultural Organization (UNESCO) [http://unesdoc.unesco.org/images/0013/001399/139949-por.pdf (accessed June 20, 2010)] shows that, between the years 1979 and 2003, *c.* 550,000 people were killed by firearms in Brazil.

To overcome this high demand of analyses, a new methodology was recently developed based on the measurements of GSR using the sector field inductively coupled plasma mass spectrometry technique (SF-ICPMS), together with ternary graphics, for results interpretation. This strategy allowed a clear identification of shooters and nonshooters (8), as well as the firearm and ammunition (9) characteristics, playing an important role, especially regarding the cases in which the used firearm has not been recovered.

In the present study, the capability of the strategy mentioned earlier to identify the type of the used firearm (revolver or pistol) in a series of test shots, using different varieties of fabrics as targets, will be demonstrated. This method may become useful during investigations involving shooting incidents, when the firearm has not been recovered.

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Materials and Methods

All experiments involving firearms were carried out at the Ballistics Laboratory of the São Paulo Criminalistics Institute. The analysis of GSR was undertaken at the Isotopic and Chemical Analysis Laboratory, Institute of Energetic and Nuclear Research. The shot tests were performed using 0.38-inch caliber revolvers, with 0.38 SPL LRN ammunition (lead round-nose cartridges) and Taurus pistols 0.40 inch S&W, supplied with S&W full metal jacket cartridges ammunition plus 9-mm caliber pistols, with Lugger FMJ Round Nose ammunition. All ammunitions used in this experiment were produced by the *Companhia Brasileira de Cartuchos (CBC; Ribeirão Pires, São Paulo, Brazil).* The choice of these firearms was based on São Paulo Police's firearm seizure statistics. The weapons were obtained from police apprehensions. All the weapons were cleaned prior to each shot test and stored in clean plastic bags. All volunteers were, previously, informed concerning the purposes of the experiments and signed a consent document before their participation. All experiments were carried out under supervision of specialists from the Ballistics Laboratory of the São Paulo Scientific Police. For safety reasons, each firearm was used only once a day and only one volunteer was admitted each time in the Ballistics Laboratory. The volunteers were chosen randomly. None of the participants were police officers, owners, or users of firearms. The targets were placed 50 cm distant from the shooters. This distance was chosen because it represents the distance similar to a great number of real cases reported by the São Paulo Scientific Police. The weapons were positioned, perpendicularly, to the fabrics. Except for security procedures, no additional guidance was given to the volunteers. Each experiment was accomplished in triplicate, and three different weapons, for each caliber, were used. Five dissimilar fabrics were used as targets: microfiber, flannel, canvas, *tergal*™ (polyester; Rhône-Poulenc, Paris, France), and *tricoline* (a variety of cotton). To avoid any contamination, both sampling procedure and the whole chemical treatment were performed in a controlled area at IPEN. The basal levels of each element in the fabrics were determined before the experiments with firearms and the values used as blank test. The analyzed areas had *c.* 2.25 cm^2 and were cut around the bullet entrance, using decontaminated pincers and scissors. Then, they were placed in polypropylene tubes, covered, and identified. The GSRs were recovered from the fabrics using 2 mL of 10% nitric acid (65% Suprapur; Merck, Frankfurt, Germany) solution, followed by 20-min agitation at 25 kHz in an ultrasonic bath (UNIQUE, Model TA1800; *CBC*), in an 80°C water bath (7).

Afterward, the extract sample solutions were diluted to 10 mL with deionized water and analyzed using an SF-ICPMS (ELEMENT 1; Finnigan MAT, Bremen, Germany), for the determination of Sb, Ba, and Pb. Working standard solutions of 1, 5, 10, 50, 100, 200, 300, and 400 μg/L in 1% nitric acid were prepared by dilution of Ba, Sb, and Pb original 1000 mg/L SPEX standards (Metuchen, NJ). Analytical curves for all analytes studied were evaluated.

To determine the total concentrations of the elements of interest, the following isotopes were measured: ^{121}Sb, ^{138}Ba, and ^{208}Pb. A Meinhard concentric nebulizer was used for sample introduction to a quartz torch, with peristaltic pumping, and 1 mL of a solution containing 10 μg/L of ^{115}In (SPEX) was used as an internal standard. The main operation conditions are shown in Table 1.

The data were interpreted using two-dimensional ternary graphs. In this type of ternary graph, the triangular coordinate systems were used to plot three variables [the components *X*, *Y*, and *Z*] in two dimensions. The points representing the relative percentage of the component variables (*Pb*, *Sb*, and *Ba*) were plotted. The diagrams were obtained using the computer code ORIGIN (31).

TABLE 1—*SF-ICPMS main operation conditions.*

Cool gas flow rate	15 L/min
Auxiliary gas flow rate	1.10 L/min
Sample gas	0.97 L/min
RF power	1300 W
Runs/passes	10/6
Wash time	40 sec
Take up time	30 sec
Sampling cone	Nickel, 1.0 mm orifice
Skimmer cone	Nickel 0.8 mm orifice
Flow rate	1.0 mL/min
Samples per peak	20
Integration window	80
Sample time	0.0100 sec
Segment duration	0.240 sec
Mass window	120
Search window	150
Scan type	Escan
Detection mode	Both
Spray chamber	Scott type (PE-Sciex)

SF-ICPMS, sector field inductively coupled plasma mass spectrometer.

Results and Discussions

Tables 2 and 3 present the concentration of the investigated elements in the fabrics, before the experiments. The samples were not submitted to any pretreatment, and these values were considered as blanks for each textile. The results obtained with the fabrics are presented in Tables 4–9. As it may be observed, there are significant differences between the concentrations of the elements of interest on the targets, before and after the shot, demonstrating, clearly, the capability of the technique to identify the investigated elements, even at very low concentrations. As expected, 0.38-inch caliber revolvers presented higher quantity of residues than the pistols. The amount of residues on the targets after shot with 0.40-inch and 9-mm caliber pistols was in the same order of magnitude.

TABLE 2—*Concentrations of metals determined from the natural fabrics before the shooting.*

Textile	Flannel			Canvas		
Element	Sb	Ba	Pb	Sb	Ba	Pb
Maximum (μg/cm^2)	0.59	6.6	13.63	0.32	5.11	6.54
Minimum (μg/cm^2)	0.01	0.1	0.31	0.01	0.12	0.31
Median (μg/cm^2)	0.24	3.22	5.76	0.1	2.67	3.14
Mean (μg/cm^2)	0.18	3.09	4.55	0.03	2.73	2.85
SD (μg/cm^2)	0.26	2.71	5.67	0.15	2.07	2.95

Number of samples: $n = 10$.

TABLE 3—*Concentrations of metals determined from the synthetic fabrics after the shooting.*

Textile	Microfiber			Tergal			Tricoline		
Element	Sb	Ba	Pb	Sb	Ba	Pb	Sb	Ba	Pb
Maximum (μg/cm^2)	0.64	1.54	1.74	0.57	3.09	7.63	0.73	5.3	8.56
Minimum (μg/cm^2)	0.01	0.11	0.32	0.02	0.14	0.54	0.01	0.11	0.29
Median (μg/cm^2)	0.37	0.82	1.12	0.24	1.33	3.69	0.4	2.38	2.77
Mean (μg/cm^2)	0.41	0.82	1.22	0.19	1.04	3.3	0.43	2.06	1.11
SD (μg/cm^2)	0.26	0.67	0.59	0.23	1.27	3.03	0.31	2.23	3.88

Number of samples: $n = 14$.

TABLE 4—*Concentrations of metals determined from the natural fabrics after the shooting.*

Firearm	0.38 revolver $n = 6$					
Textile	Flannel			Canvas		
Element	Sb	Ba	Pb	Sb	Ba	Pb
Maximum (μg/cm^2)	98.98	109.17	3191.02	39.94	37.86	1088.91
Minimum (μg/cm^2)	4.28	8.95	127.04	2.04	10.4	86.9
Median (μg/cm^2)	41.35	42.2	1463.88	17.49	21.82	480.31
Mean (μg/cm^2)	30.16	35.51	1433.98	9.59	17.21	265.12
SD (μg/cm^2)	39.17	37.86	1255.36	19.73	14.3	534.54

n, number of samples.

TABLE 5—*Concentrations of metals determined from the natural fabrics after the shooting.*

Firearm	0.40 pistol $n = 6$					
Textile	Flannel			Canvas		
Element	Sb	Ba	Pb	Sb	Ba	Pb
Maximum (μg/cm^2)	18.48	17.61	78.53	40.87	46.51	132.73
Minimum (μg/cm^2)	1.45	3.6	9.52	0.13	4.31	2.62
Median (μg/cm^2)	7.99	8.06	38.09	10.61	15.02	38.79
Mean (μg/cm^2)	6.77	6.92	34.33	2.84	8.95	17.49
SD (μg/cm^2)	7.22	5.5	28.96	16.16	16.16	50.91

n, number of samples.

TABLE 6—*Concentrations of metals determined from the natural fabrics after the shooting.*

Firearm	9 mm pistol $n = 6$					
Textile	Flannel			Canvas		
Element	Sb	Ba	Pb	Sb	Ba	Pb
Maximum (μg/cm^2)	109.23	92.88	471.8	8.36	33.24	142.49
Minimum (μg/cm^2)	0.17	0.2	0.4	0.11	5.24	1.65
Median (μg/cm^2)	20.3	22.38	90.43	1.49	12.09	37.23
Mean (μg/cm^2)	8.36	9.3	31.26	0.2	8.55	9.67
SD (μg/cm^2)	30.76	26.32	132.44	2.68	9.5	48.33

n, number of samples.

A careful observation of the data presented in Tables 4–9 shows a wide distribution range of elemental concentrations on the fabrics. A significant variation among the types of fabrics was not found. Neither the mean nor the median values showed consistency, making any possible interpretation difficult, based only on these values. In fact, with basis on this study strategy, neither the use of the mean nor the median value should be used, with the objective of establishing a recovery criterion for the residues. The experiments were planned in an attempt to reproduce real situations, where firearms are used at a short distance, as it occurs with the majority of

TABLE 7—*Concentrations of metals determined from the synthetic fabrics after the shooting.*

Firearm	0.38 revolver $n = 9$								
Textile	Microfiber			Tergal			Tricoline		
Element	Sb	Ba	Pb	Sb	Ba	Pb	Sb	Ba	Pb
Maximum (μg/cm^2)	32.64	20.67	973.88	20.79	29.45	492.8	63.43	72.04	1597.58
Minimum (μg/cm^2)	0.82	1.49	16.22	0.8	1.54	8.09	0.8	2.07	37.65
Median (μg/cm^2)	6.5	6.58	194.87	4.97	10.37	125.22	14.86	20.33	413.31
Mean (μg/cm^2)	3.24	5.16	117.94	2.81	8.34	70.42	3.36	11.18	95
SD (μg/cm^2)	9.01	5.9	276	6.28	8.92	152.16	22.53	23.48	593.12

n, number of samples.

TABLE 8—*Concentrations of metals determined from the synthetic fabrics after the shooting.*

Firearm	0.40 pistol $n = 9$								
Textile	Microfiber			Tergal			Tricoline		
Element	Sb	Ba	Pb	Sb	Ba	Pb	Sb	Ba	Pb
Maximum (μg/cm^2)	64.17	58.64	200.64	55.94	146.04	166.5	21.76	17.13	64.61
Minimum (μg/cm^2)	0.18	0.33	1.18	0.43	0.99	1.39	5.34	5.21	13.66
Median (μg/cm^2)	8.6	11.45	30.67	12.41	23.21	56.02	12.55	10.01	35.11
Mean (μg/cm^2)	2.49	6.03	18.04	5.65	10.87	42.68	10.54	7.7	27.06
SD (μg/cm^2)	16.18	14.73	49.79	16.37	39.21	56.93	8.39	6.29	26.41

n, number of samples.

TABLE 9—*Concentrations of metals determined from the synthetic fabrics after the shooting.*

Firearm	9 mm pistol $n = 9$								
Textile	Microfiber			Tergal			Tricoline		
Element	Sb	Ba	Pb	Sb	Ba	Pb	Sb	Ba	Pb
Maximum (μg/cm^2)	26.96	63.11	103.81	66.04	68.16	233.76	18.91	19.39	48.05
Minimum (μg/cm^2)	0.19	0.72	1.37	0.57	5.86	5.73	0.42	2.51	2.34
Median (μg/cm^2)	5.43	9.09	22.23	11.61	22.87	76.43	5.68	7.41	15.32
Mean (μg/cm^2)	2.85	2.54	7.51	3.08	12.77	50.38	2.94	5.08	9.19
SD (μg/cm^2)	6.81	16.31	27.99	19.26	19.46	80.72	7.18	6.51	16.93

n, number of samples.

homicide cases faced by the Scientific Police of São Paulo. It is well known that the distribution of GSR on targets depends on several factors, such as the distance between the firearm and the target, the state of conservation of the firearm, the type and the state of conservation of the target, the presence of residues from other sources on the target, environmental conditions, the bullet penetration angle, and others. Thus, as expected, the amounts of the recovered residues from the fabrics were dependent on the variation from shot to shot, for each firearm and for each volunteer. Therefore, the presence of these metals on the targets should be interpreted with caution to avoid false positives or false negatives, detected with colorimetric tests, commonly used by the São Paulo State Police or even using spectroscopic techniques, as reported in the literature.

However, the amounts of metals present in the targets were not completely random. The use of ternary graphs might be an important tool for the interpretation of these data. The results are presented in Figs 1–4. A given point, at one of the triangle vertices, indicates that the relative concentration of the corresponding component, at that point, is 100%. In contrast, a given point, exactly in the center of the triangle, shows that the relative percentage of the three components is equal. The data are automatically normalized when the diagrams are created.

As it may be seen, the residues show a clear distribution pattern allowing samples used as targets to be distinguished from those used as blank samples. A characteristic pattern of distribution for pistols and round-barrel revolvers could, also, be observed. The reason of this behavior is not completely clear at this point of our research. One hypothesis for this distribution pattern is likely to be related to the shot energy. The shot energy for 0.40 in S&W and the 9-mm Lugger pistols are similar, 344 and 374 ft-lb, respectively, while for 0.38 in SPL LRN is 235 ft-lb. As it can be observed in Fig. 2, the majority of the results can identify positively testimonies from revolvers.

The presence of the elements of interest in the bullet, bullet coating or jacket, plus cartridge components do not make any

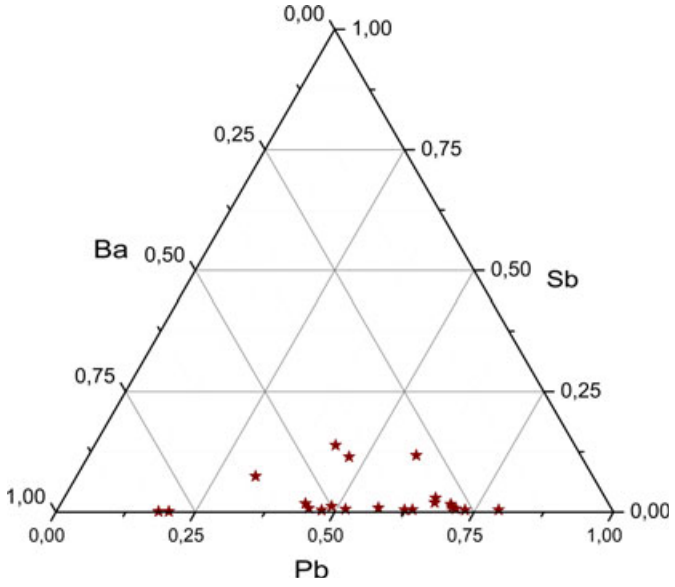

FIG. 1—*Ternary graph of the distribution of metals from blank samples of fabrics.*

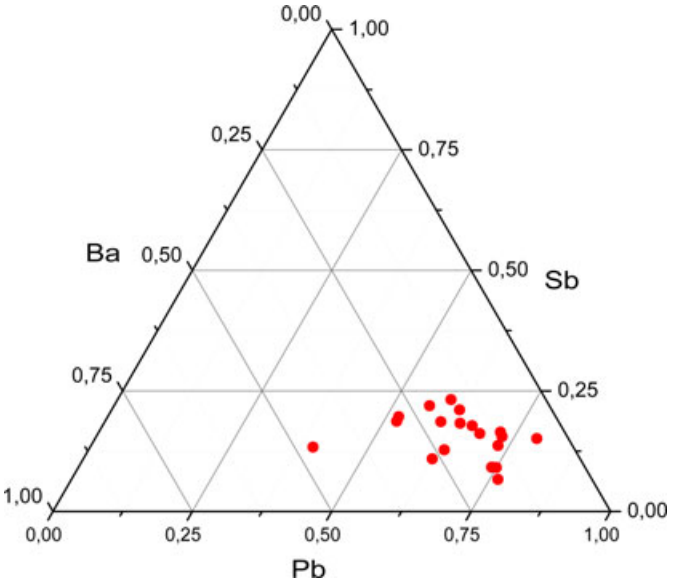

FIG. 3—*Ternary graph of the distribution of metals after shots in fabrics with a 0.40 inch caliber pistol.*

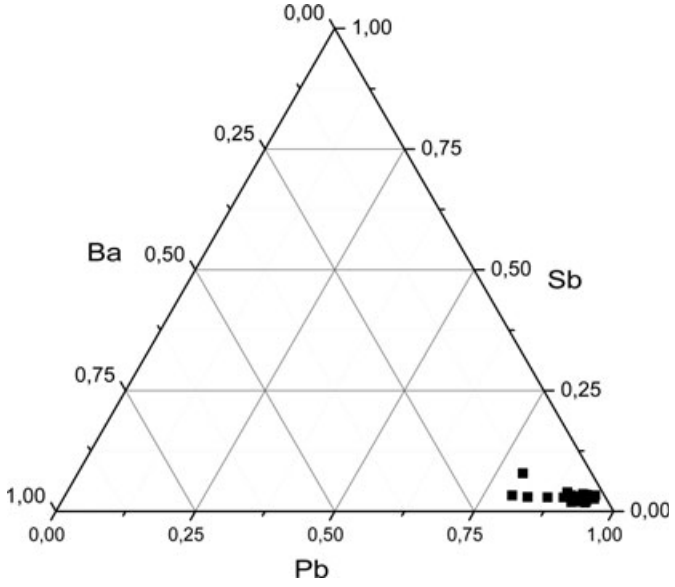

FIG. 2—*Ternary graph of the distribution of metals after shots in fabrics with a 0.38 inch caliber revolver.*

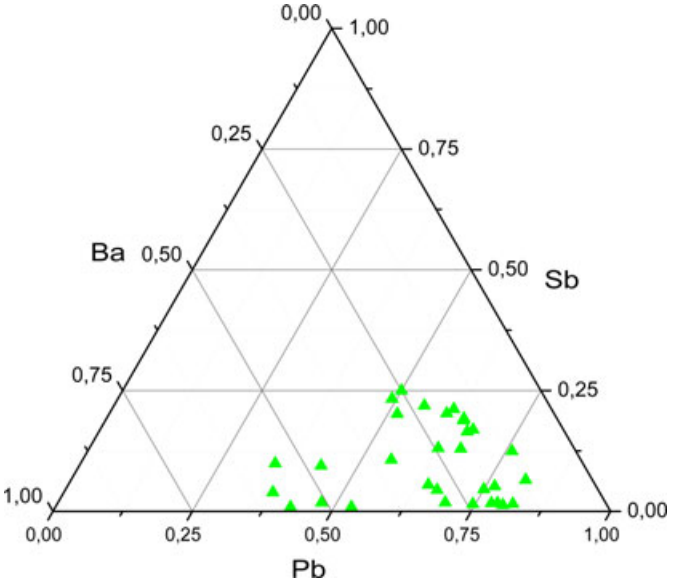

FIG. 4—*Ternary graph of the distribution of metals after shots in fabrics with a 9 mm caliber pistol.*

difference in the patterns of distribution obtained with the ternary graphics. As a consequence, the investigated elements show different graphic representations for revolvers and pistols, which have a similar and more dispersed distribution.

Conclusions

The present work demonstrated the capability of a high-sensitive technique, like SF-ICPMS, to determine, at ultratrace levels, GSR on different types of fabric targets, after a shot. It was possible to quantify the elements of interest (Pb, Ba, and Sb) deposited in a small area around the bullet entrance hole. The quantitative determination of the metals is not relevant, because the amount of the investigated elements may be altered owing to several reasons.

The use of a region not directly exposed to the residues, as a blank sample, was very useful for data interpretation, allowing a clear origin identification of the metallic elements present on the studied samples.

The use of ternary graphs permitted to identify specific patterns of distribution for blank samples and a patent distinction between revolvers and pistols.

This approach may be used in real cases, analyzing pieces of the fabrics collected around the bullet entrance hole and comparing these results, by the use of ternary graphics, with other pieces collected in different parts of the victim clothes. The difference among

patterns of distribution may be employed as preliminary screening on the use and type of firearm.

Considering the high sensitivity of the used technique, its significant sample throughput and the employment of a small piece of evidence for the analysis, leaving enough material to be analyzed by other methodologies, it showed to be suitable for use in a scenario of high analysis demand.

The patterns of distribution found in the fabrics demonstrated to be very similar to those obtained in the shooters' hands (7–9); the use of ternary graphs might be very helpful during the confrontation of evidence involving pieces of clothing and possible crime suspects. The described method is very promising; however, new experiments have to be conducted to establish its robustness.

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