

Submitted: 15 July 2026

Accepted after peer review: 20 August 2026

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◆ Research article

Determination of the Muzzle-to-Target Distance in Shooting Cases Using Inductively Coupled Plasma – Optical Emission Analysis of Barium, Lead, and Antimony

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Keywords: gunshot residue, IGSR, ICP-OES, muzzle-to-target distance, barium, lead, antimony, forensic ballistics, shooting distance, polynomial regression

Abstract

Between 2015 and 2023 firearm-related deaths almost doubled in the U.S., increasing discussions concerning gun control measurements. Gun regulations vary between states. States like Mississippi have not only relaxed gun control laws but also the highest firearm-related deaths. Research involving inorganic gunshot residue (IGSR) typically focuses on the shooter end rather than the target material. This work aimed to create a mathematical relationship between the concentration of metals on target materials and the muzzle-to-target distance for the estimation of shooting distance. Cloth samples containing IGSRs were obtained by shooting a Remington UMC 9mm Luger ammunition from a Smith & Wesson M&P9 Shield semi-automatic pistol in a controlled indoor environment at varying distances. The IGSR was extracted from samples using an acid digestion procedure and analyzed using Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) to quantify barium, lead, and antimony. A mathematical model was developed for the interpretation of barium, lead, and antimony in both ammunitions analyzed. Polynomial regression was employed as an exploratory tool to visualize the non-linear relationship between IGSR concentration and shooting distance within the experimental range studied. This research provides evidence that the relationship between muzzle-to-target distance and the concentration of metal on the target material not only exists but is polynomial. The results demonstrate a measurable non-linear relationship between inorganic gunshot residue deposition and shooting distance. While additional validation across different firearms, ammunition types, and target materials is required,

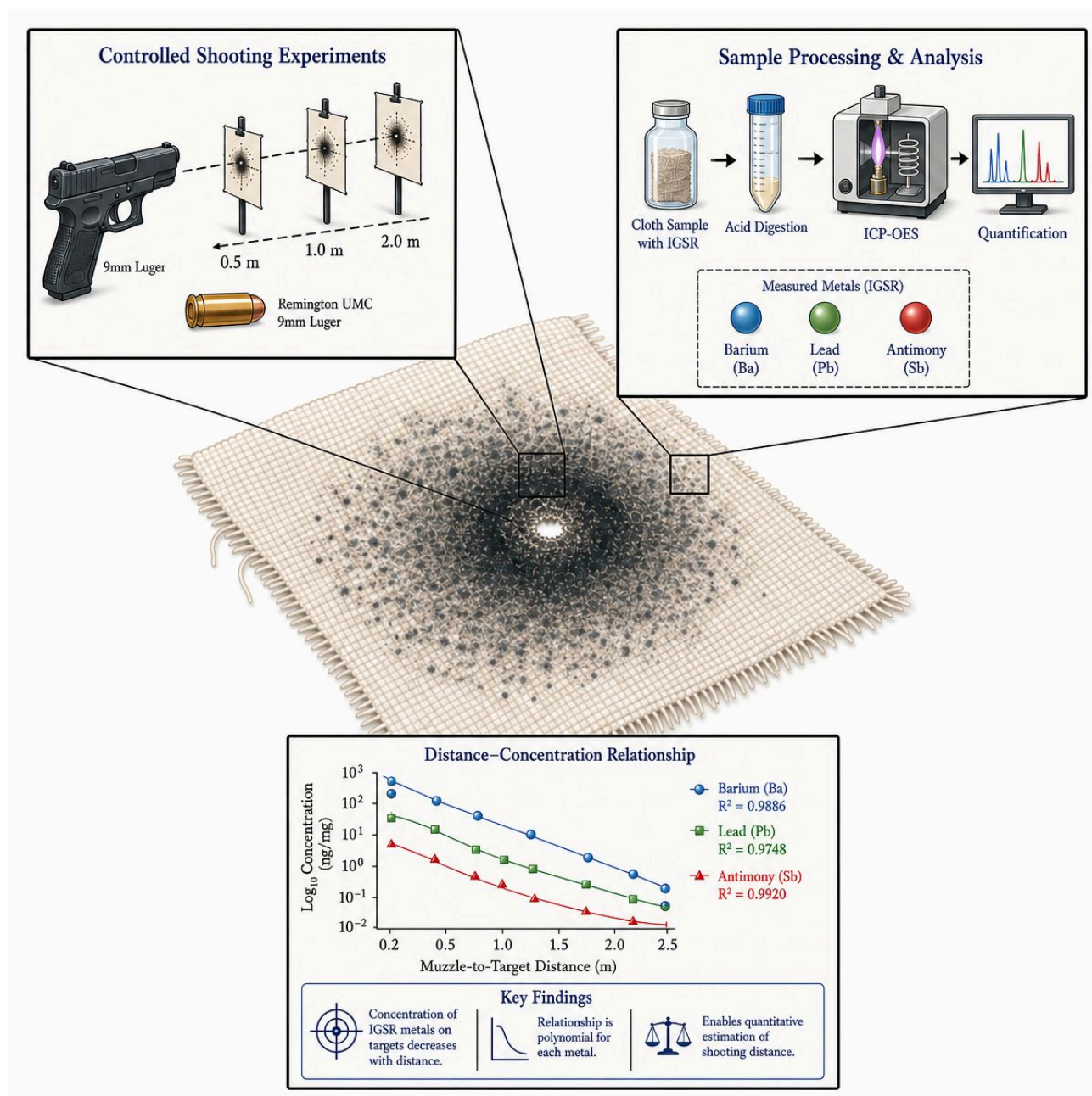
these findings provide evidence supporting the future development of quantitative approaches for shooting distance estimation.

Introduction

Gun violence is a leading cause of premature death in the U.S., with 9,839 firearm-related deaths reported in July 2026 [1]. Considering the criminal justice perspective, the accurate reconstruction of events and prompt apprehension of individuals involved in these crimes is critical. Thus, firearms and other firearm-related evidence are crucial to criminal investigations. Forensic ballistics is the science of the motion of projectiles and represents a sub-area of criminalistics that includes the study of firearms and ammunition [2]. The shooting of a biological or other solid target with a firearm generates a complex and informative pattern of evidence, comprising of impacts, traces, and residues that emerge from the firing of the gun and the interactions of projectile, target material, and surrounding environmental conditions. One of the most important transfers of evidence found after the discharge of a firearm is called gunshot residue (GSR), and it is frequently used to elucidate important information concerning the nature of the gun, the distance of the shot, the angle, and much more. GSR is mainly composed of burnt, partially burnt, and unburnt organic or inorganic particles from the explosive primer from the cartridge, the propellant, and possibly fragments of the bullet, cartridge case, and even the firearm, that will frequently contain antimony (Sb), barium (Ba), and lead (Pb) or zinc (Zn), copper (Cu), and tin (Sn) [3]. They can be recovered not only from the hands and clothes and other parts of the shooter but also from the victim and/or target material struck by the projectile [4]. When correctly analyzed, GSR can yield information about the type of ammunition, the shooting distance, and the spatial position of the discharged firearm in relation to its surroundings and target. The chemical aspect of the GSR can be extremely informative as it varies with the type of ammunition. Currently, many techniques such as microscopy, scanning electron microscopy, energy dispersive X-ray spectroscopy, flameless atomic absorption spectroscopy, and energy dispersive X-ray fluorescence [4–7] are used for the detection of GSR in potential suspects. Colorimetric testing is usually applied to estimate the shooting distance, often referred to as the muzzle-to-target distance. The Griess or Modified Griess test (MGT) is used to detect the presence of nitrites using a colorimetric reaction captured on chemically treated photographic paper [8]. The Sodium Rhodizonate test is used to detect the size and relative density of the lead residues present in a GSR pattern [9]. Both chemical tests are qualitative and, therefore, lead to severely and scientifically limited estimation ranges being reported. Current and previous research has consistently focused on collecting data and samples from the shooter, as opposed to the target material. This traditional approach to GSR detection has increasingly met court challenges as to the limited probative value of the detection results and many possible sources of individual alleged GSR particles. By focusing on the target material, a more complete GSR profile can be explored on a surface in which the origin of the residues is not in question. Conventional ammunition is formed by a primer that comprises primarily lead styphnate, barium nitrate, and antimony sulphide, being the presence of Ba, Pb, and Sb, characteristics of a GSR [10]. Despite modern ammunition being produced by different elemental compositions, for example, lead-free ammunition [11], the most popular ammunition in the market is still conventional. Thus, the aim of this work was to establish a mathematical relationship to determine muzzle-to-target shooting distance by quantitative analysis of IGSRs on target material using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-

OES). This technique is a powerful tool for rapid analysis of chemical elements due to its high sensitivity, selectivity, and multi-elemental character [10,12–14] being successfully applied in different forensic analysis.

An ICP-OES method for the analysis of Ba, Pb, and Sb was developed and validated for the quantification of these elements to create a mathematical relationship between their concentration and the shooting distance.



Graphical abstract

Methods

Reagents and Standards

All reagents used were analytical grade. Solutions were prepared using ultrapure water (resistivity 18.2 MΩ/cm) obtained from a Direct-Q3 water purification system (Millipore Corporation, Burlington, MA, USA). Trace metal grade concentrated nitric acid (67-70%) and concentrated hydrochloric acid (34-37%) were obtained from Fisher Scientific (Waltham, MA, USA). The HNO₃ was diluted in ultrapure water to produce the 50% (v/v) washing solution and 5% (v/v) for dilution of standards and blanks. A 30% hydrogen peroxide solution was obtained from Sigma Aldrich (St. Louis, MO, USA) and stored at 4°C before use. Filter paper (7.0 cm and 11.0 cm) was obtained from Fisher Scientific (Waltham, MA, USA). Anhydrous ethanol for cleaning was obtained from Koptec (King of Prussia, PA, USA). Individual elemental stock solutions containing 1000 ppm of Ba, Pb, and Sb (Inorganic Ventures, Christiansburg, VA, USA) were applied to prepare the calibration curves and the QA/QC standards. Yttrium was used as the internal standard at a concentration of 2 ppm in all standards, samples, and blanks. A 100 ppm yttrium stock solution was purchased from Inorganic Ventures (Christiansburg, VA, USA). Argon 99.999% pure (AirGas, Radnor, PA, USA) was used as auxiliary gas, to ignite the plasma, and to purge the optics for the ICP-OES analysis.

Shooting Procedure

All shootings were performed in the indoor firing range at the Virginia Commonwealth University Police Department. A Smith & Wesson Model M&P9 Shield, caliber 9 mm Luger, semi-automatic pistol was utilized for all samples. Prior to each shooting session, the firearm was cleaned using generic firearm cleaning materials (RemOil cleaning solution, cotton swabs, and swatches). Shooting QCs were established to guarantee no cross-contamination from previous shootings. For that, a blank white cloth was left in the range during the procedure and another white cloth was stored before the shooting. To stabilize the firearm during the shooting, a gun vise was set on a table. To adjust distance, the gun vise was moved accordingly, and a standard measuring tape was used for verification (Figure 1). The target material consisted of a standard 9×9 inch white cotton twill jean cloth. Each cloth was stapled to a piece of cardboard, then stapled to a larger piece of cardboard that was clipped in place within the shooting lane for further stabilization. The ammunition tested in this study was the Remington UMC, caliber 9 mm Luger, 115 grain FMJ. The shooting procedure was performed at contact (0), 3, 6, 12, 15, 18, 21 and 24 inches. Each pattern produced from an individual shot was then collected by packaging the white cloth in a high-quality evidence-grade plastic bag which was sealed until ready for sample preparation.



Figure 1. (a) Shooting vise setting using the Smith & Wesson M&P9 Shield, caliber 9 mm Luger, semi-automatic pistol used to obtain all samples. (b) Demonstration of measurement from the muzzle of the firearm to the center of the target material at 6 inches. The same set was applied for all distances tested.

Sample Preparation and Analysis

Four circles of 9/16 inches in diameter were collected from a 2-inch distance from the center of the shooting target in four directions (north, south, east, and west) using a homemade plexiglass template as shown in Figure 2. Circles were collected using a clean, sterile scalpel. All four circles of the same sample were weighted and digested together. Samples and the QCs were acid-digested according to a modified EPA procedure [15]. For the digestion, 0.1 g of each sample (equivalent to 4 circles) was added to a clean 50-mL beaker, followed by 1 mL of 50% (v/v) nitric acid. Beakers were heated covered at $95^{\circ}\text{C} \pm 5$ for 15 minutes without boiling. Five hundred μL of concentrated nitric acid were added, and the samples were heated at the same temperature for 30 minutes and repeated three times until the brown fumes disappeared. Watch glasses were removed, and the solutions were evaporated to 1 mL, and allowed to cool. Next, 200 μL of ultrapure water and 300 μL of cold 30% (v/v) H_2O_2 were added, and samples were heated slightly at $95^{\circ}\text{C} \pm 5$ until effervescence subsided. Then, samples were covered, and 1 mL of concentrated HCl was added and heated again for 15 minutes. The solution was removed from the heat and allowed to cool, then filtered using a paper filter. The final solutions were quantitatively transferred to 25-mL volumetric flasks and diluted with 5% (v/v) nitric acid to a final volume of 25 mL. Finally, samples were filtered, spiked with the internal standard at a final concentration of 2 ppm, and stored for analysis. Samples and shooting QCs were prepared in triplicate ($n=3$).

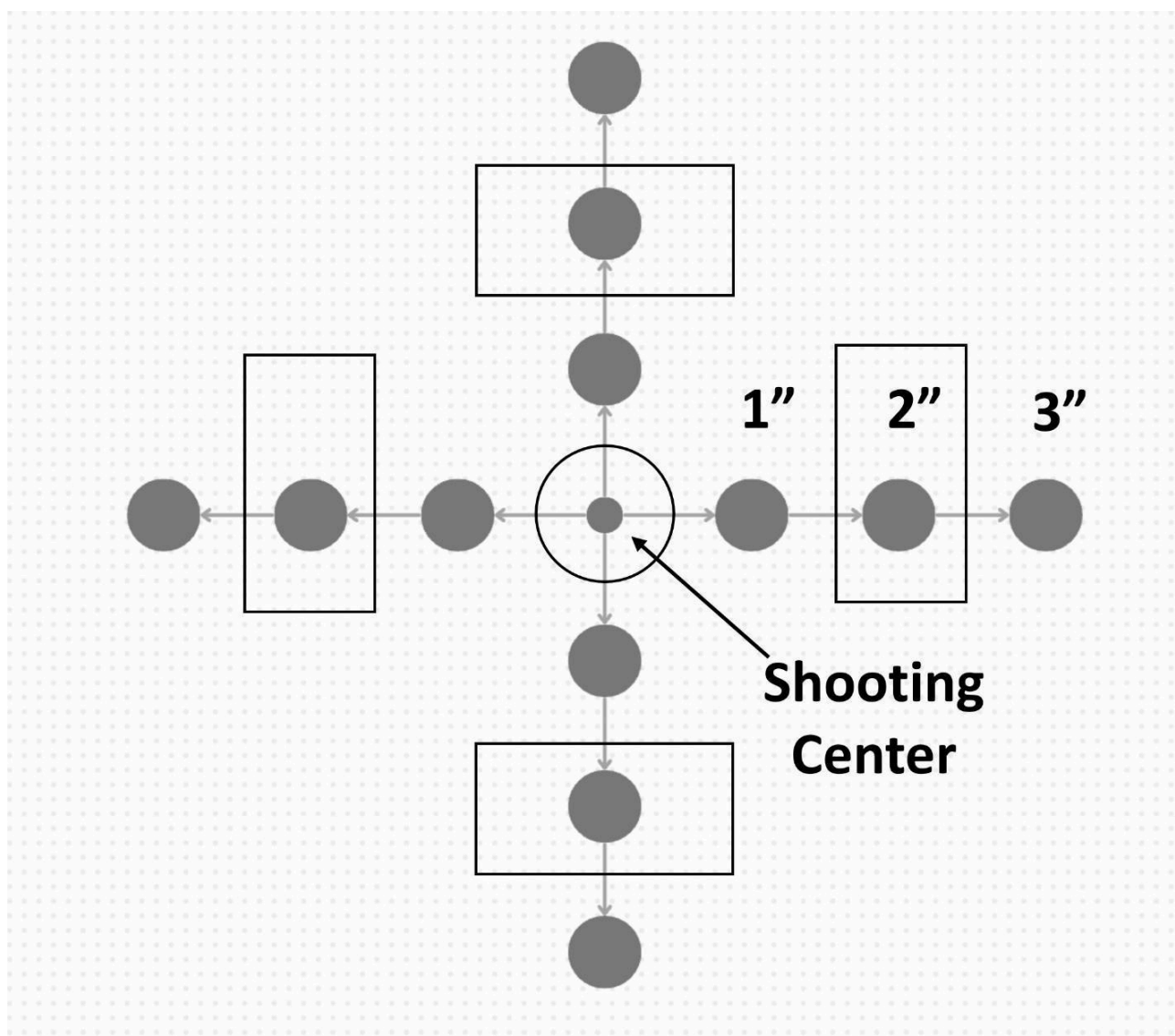


Figure 2. Plexiglass scheme for the collection of the GSR samples after shooting. The collection was performed 2" from the shooting center, in all four directions as marked with the rectangles.

ICP-OES conditions

An Agilent 5110 inductively coupled plasma optical emission spectrometer (Santa Clara, CA, USA), with an Agilent SPS 4 autosampler was used for the simultaneous determination of Ba, Pb, Sb, and Y in digested GSR samples. The following wavelengths (in nm) for each element were selected as the ones with less spectral interference according to previous analysis performed by our group: 455.403 Ba; 220.353 Pb; 206.834 Sb; and 371.029 Y. The ICP-OES was operated with a RF power of 1.40 kW, plasma flow rate of 12.0 L/min, auxiliary gas flow rate of 1.00 L/min, and nebulizer flow rate of 0.60 L/min. The plasma was positioned in radial viewing mode. The samples were introduced into the plasma via the nebulizer.

Method Validation

The method was validated according to the EPA Method 6010D for ICP-OES [16] and the ANSI/ASB 036 Standard Practices for Method Validation in Forensic Toxicology [17]. The evaluated parameters

were sensitivity, linearity, limit of detection (LOD), limit of quantification (LOQ), accuracy, recovery, and precision. For the evaluation of bias and precision, standard reference solutions for Ba, Sb, and Pb at low, medium, and high concentrations were prepared at concentrations of 0.5 ppm, 3.5 ppm, and 5.5 ppm, respectively. Precision was evaluated as repeatability (intra-day) and reproducibility (inter-day). The acceptance criteria for bias and precision were according to the ANSI/ASB 036 Standard Practices for Method Validation in Forensic Toxicology which states that the maximum acceptable bias shall be $\pm 20\%$ at each concentration. For precision, %CV (coefficient of variation) should not exceed 20% at each concentration. The limit of detection (LOD) was also assessed by running a series of six very low concentration samples (0.05 ppm). The LOD is defined as $3 \times s/m$, where s is the standard deviation of all the very low concentration measurements and m is the slope of the respective calibration curve. Recovery and accuracy were evaluated by analyzing white clothes spiked with 0.5, 3.5, and 5.5 ppm of the standards, followed by the extraction procedure and analysis.

Data Analysis

Statistical analysis of all results was performed using Microsoft Excel and GraphPad Prism 9.1.2. The Grubbs Test was used to determine the presence of outliers. Differences between the two groups were calculated by unpaired t-tests. For all tests, a p-value of less than 0.05 was considered statistically significant.

Results

Method Validation

The developed method proved to be linear in the range of 0.2–6 ppm for all analytes. Calibration curves for each target element were analyzed in triplicate and averaged over three sequential days. Linearity was achieved for all standards as showed by R^2 values exceeding 0.998 for all elements. The linearity range was chosen considering previous literature [18]. The limit of detection values varied from 0.001 to 0.062 ppm, respectively. The limit of quantification was considered the first point of the calibration curve at 0.2 ppm for all elements. The %CV varied from 0.07 to 5.6% for the intra-day precision, and from 1.0 to 6.4% for inter-day precision. For the accuracy, all %CV varied from -8.69 to 3.65%. All elements results were in accordance to the ANSI/ASB 036 Guideline acceptance parameters for accuracy and precision [17]. Results showed that the acid-digestion method showed satisfactory results for GSR sample preparation for ICP-OES analysis. The average recovery (%) of Ba, Pb, and Sb were 98.6, 98.5 and 99.7%, respectively. The results for all validation parameters obtained are shown in Table 1.

Element	n=3	y = mx + b	Concentration Range (ppm)	R ²	LOD (ppm)	LOQ (ppm)	QC conc. (ppm)	Intraday Precision (% n=3)	Interday Precision (% n=3)	Bias (% n=3)	Recovery (% n=3)
Barium	Day 1	y=4.45x+0.16	0.2-6	0.9999	0.001	0.200	0.5	0.314	1.055	-7.50	92.50
	Day 2	y=4.37x+0.3	0.2-6	0.9995			3.5	0.070	1.071	1.45	101.45
	Day 3	y=4.47x+0.12	0.2-6	0.9999			5.5	0.141	1.761	1.95	101.95
Lead	Day 1	y=0.01131x+0.0008	0.2-6	0.9999	0.062	0.200	0.5	0.375	1.010	-8.69	91.31
	Day 2	y=0.01099x+0.0011	0.2-6	0.9997			3.5	0.868	1.503	1.62	101.62
	Day 3	y=0.01144x+0.0006	0.2-6	0.9999			5.5	0.382	1.619	1.65	101.65
Antimony	Day 1	y=0.0061x+0.0007	0.2-6	0.9986	0.047	0.200	0.5	1.227	6.373	-4.84	95.16
	Day 2	y=0.0061x+0.0006	0.2-6	0.9998			3.5	5.590	1.111	0.40	100.40
	Day 3	y=0.0063x+0.0002	0.2-6	0.9998			5.5	2.249	1.991	3.65	103.65

Table 1. Figures of Merit for the Validated Method. Parameters of the Analytical Curves of Barium, Lead, and Antimony (0.2–6 ppm) were obtained by the Least Squares Method.

Quality Control of the Shooting Range

To guarantee absence of cross-contamination, two QC for the shooting range were prepared. A blank sample present in the shooting range during the procedure (blank on range), and a blank sample stored before the procedure (blank off range). The results are shown in Figure 3. The analysis showed that the amount of Ba, Pb, and Sb, in both blank controls were statistically different from the samples obtained by shooting the Remington UMC 9 mm Luger from a distance of 6 inches, showing no detectable concentrations of the elements analyzed. Thus, the shooting range was suitable for the procedure and no cross-contamination was detected.

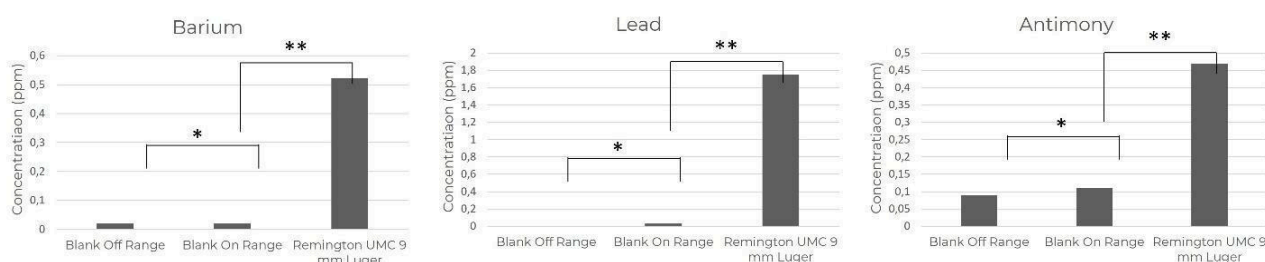


Figure 3. Analysis of the concentration of barium, lead, and antimony in range off, range on, and samples for the evaluation of cross-contamination during the shooting procedure. Values are given as mean $\pm$ S.E.M (n=9). *p < 0.05; **p < 0.02.

Barium, Lead, and Antimony for Shooting Distance Correlations

The analysis of the correlation between the muzzle-to-target distance and concentration of the metals found on the target material are shown in Figure 4a–c. The relationships between the elements analyzed showed no linear correlation but a polynomial function with a wave-like graphic representation. Therefore, these relationships indicate that as a shooter moves further away, the concentration of residue on the target material does not decrease linearly but instead fluctuates. The observed concentration profiles suggest that interpretation based on a single elemental marker may be ambiguous at certain distances and that the combined evaluation of multiple elemental concentrations may provide improved discriminatory power.

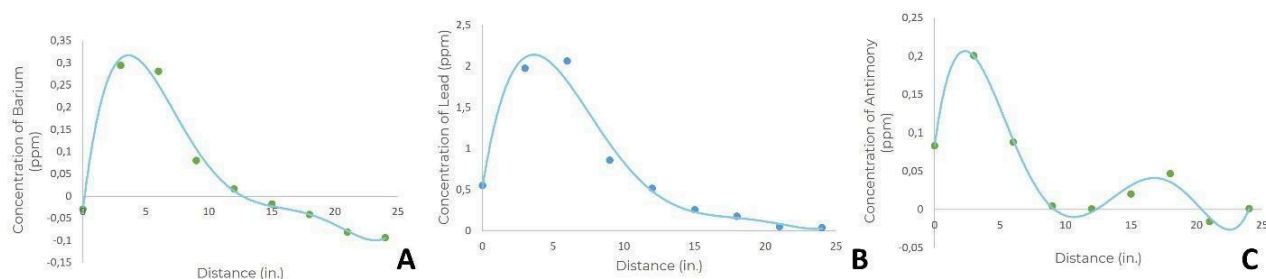


Figure 4. Relationship between muzzle-to-target distance and concentrations of (A) barium, (B) lead, and (C) antimony. Points represent mean concentrations obtained at each shooting distance. The fitted curves are smoothing functions intended to aid visualization of the observed trends and should not be interpreted as predictive models. Graphical representation of the polynomial mathematical relationship of the Ba, Pb, and Sb concentrations and the shooting distances. (a) Barium analysis ($y = 2 \times 10^{-6} x^5 - 0.0002 x^4 + 0.0044 x^3 - 0.0527 x^2 + 0.2369 x - 0.0327$, $R^2 = 0.9886$); (b) Lead analysis ($y = 8 \times 10^{-6} x^5 - 0.0006 x^4 + 0.0182 x^3 - 0.231 x^2 + 1.0726 x + 0.5271$, $R^2 = 0.9748$); (c) Antimony analysis ($y = 3 \times 10^{-6} x^5 - 0.0002 x^4 + 0.0041 x^3 - 0.0392 x^2 + 0.1243 x + 0.0832$, $R^2 = 0.992$). Graphs are represented as element concentration in ppm vs the shooting distance in inches.

Discussion

The Gunshot Residue Subcommittee of the Organization of Scientific Area Committees (OSAC) for Forensic Science classifies particles as one of the following: "characteristic of", "consistent with", or "commonly associated with" GSRs. Particles that are classified as characteristic of GSR contain lead (Pb), barium (Ba), and antimony (Sb). These three elements typically originate from ammunition that uses primers based on the "Sinoxid" formula which contains a mixture of lead styphnate as an explosive, barium nitrate as an oxidizer, and antimony sulfide as fuel. Some GSR particles may only contain two out of these three elements. For this reason, the determination of barium, lead, and antimony concentrations may provide complementary information regarding shooting distance. These elemental profiles may be interpreted individually or, preferably, in combination to generate a more comprehensive assessment of muzzle-to-target distance when conventional ammunition is used [19].

The ammunition chosen for this study was the Remington UMC 9 mm Luger FMJ. This ammunition is not lead-free and therefore, the analysis for the distance and concentration relationship was conducted on lead, barium, and antimony. The relationships observed between elemental concentration and shooting distance exhibited non-linear patterns characterized by local maxima and minima [20]. The pattern observed in all correlations may be related to the behaviour of the expanding turbulent muzzle gas plume generated during the firearm discharge. The "expanding turbulent muzzle gas plume" is a distinctive mushroom-shaped cloud of debris, smoke, and condensed water resulting from an explosion [21,22]. Even mostly associated to nuclear explosions or volcanic eruptions, the mushroom cloud is also observed in gunshots and can define the lack of linearity of the correlation of the concentration of the residues and the shooting distance. This occurs because they result from the sudden formation of a large volume of lower-density gases nearby the gun causing a Rayleigh–Taylor instability [23,24]. The gases then move rapidly, resulting in turbulent vortices curling downward around their edges, forming a temporary vortex ring that draws up a central column that creates the direction of the bullet [22]. Because of this phenomenon, different waves of elements present in the ammunition will accumulate in the target in different patterns, creating residual profiles that are not linear. Therefore, these relationships indicate that as

a shooter moves further away, the concentration of residue on the target material does not decrease linearly but instead fluctuates. Depending on the proximity or distality of the shooter, this effect may change or appear differently. For example, at contact (0 inches), there is no room for the mushrooming to begin and, therefore, high concentrations of residue are found on the target material as shown by the results of this study. This effect is the cause of a nonlinear relationship between muzzle-to-target distance and concentration of metals found in target material after discharge [25].

The analysis of lead concentrations leads to an asymptotic function which resembles a more linear relationship after 10 inches of distance. Lead is an element found in multiple parts of a cartridge, such as the primer and the projectile, whereas barium and antimony are solely found in the primer residue. This could potentially cause lead components to behave differently such as depletion over a further distance. Lead appears to approach a concentration of 0 ppm around 24 inches, whereas barium depletes at approximately 12 inches, and antimony around 9 inches. The relationship for antimony appears to have a secondary curve with an increase of concentration around 12 inches. This could be a result of the expanding turbulent muzzle gas plume causing an increase in antimony at this distance in this particular ammunition. Additionally, given that the trend for antimony appears to have multiple peaks, there is a possibility that when utilizing antimony for the muzzle-to-target distance calculation, there would be multiple possible values.

Although individual elemental profiles exhibited non-monotonic behaviour, forensic interpretation would not be expected to rely on a single analyte. Instead, the combined concentrations of Ba, Pb, and Sb may be evaluated simultaneously to generate an integrated elemental profile associated with a particular shooting distance. In practical casework, the simultaneous assessment of multiple elemental markers would be expected to provide greater discriminatory power than the interpretation of a single concentration value alone. For example, while a measured antimony concentration may correspond to more than one possible distance because of the presence of local maxima and minima, the corresponding concentrations of barium and lead could substantially narrow the range of plausible distances. Consequently, future distance-estimation models may benefit from the application of multivariate statistical approaches capable of integrating multiple elemental measurements simultaneously. Techniques such as principal component analysis (PCA), partial least-squares (PLS) regression, machine-learning regression algorithms, or Bayesian likelihood-based models could be used to establish probabilistic relationships between elemental concentration patterns and muzzle-to-target distance. Under such an approach, the complete elemental signature deposited on the target material would be evaluated rather than individual elemental concentrations, thereby reducing uncertainty associated with local maxima or minima observed in single-element distributions. Such multivariate approaches are widely employed throughout analytical chemistry and forensic toxicology to improve classification and predictive accuracy when multiple variables contribute to the observed outcome [26–28]. Therefore, the concentration profiles described in the present study may provide the foundation for future predictive models capable of providing more robust and reliable estimates of shooting distance than models based on individual elemental concentrations alone.

Conclusion

Herein, we provide a validated ICP-OES for the analysis of barium, lead, and antimony in gunshot samples for the evaluation of muzzle-to-target distance. A non-linear, distance-dependent relationship between shooting distance and concentration of barium, lead, and antimony deposited on target materials was observed. The present dataset represents an initial proof-of-concept investigation and additional sampling distances should be included to better characterize the relationship between elemental concentration and muzzle-to-target distance and to improve the robustness of future predictive models. As this study only evaluated distances within 2 feet, once a more established profile is obtained, further distances should be included. This process should be repeated with other ammunition types, including lead-free ammunition. Furthermore, the Modified Griess Test and Sodium Rhodizonate Test should also be used in conjunction with this study to determine if past techniques are both consistent and reliable, given new knowledge of the general correlation of the concentration of IGSR on target material. Several factors may influence IGSR deposition patterns, including ammunition formulation, primer composition, barrel length, firearm design, target substrate, weapon condition, and environmental variables such as airflow and humidity. The present study was intentionally designed as a proof-of-concept investigation employing a single firearm model, a single ammunition type, and a single cotton-based target material under controlled indoor conditions. Consequently, the concentration-distance relationships observed herein should not be interpreted as universally applicable. Future work should systematically evaluate additional firearms, ammunition formulations, target materials, and environmental conditions to establish the robustness and transferability of the observed concentration-distance relationships.

An important observation of this study is that individual elemental concentrations may not uniquely define a shooting distance due to the presence of local maxima and minima in certain elemental profiles, particularly for antimony. Consequently, future forensic applications will likely require multivariate approaches that integrate the combined concentrations of barium, lead, and antimony rather than relying on individual elemental measurements. The development of models based on multivariate statistical approaches, such as principal component analysis (PCA), partial least-squares (PLS) regression, machine-learning algorithms, or Bayesian frameworks, may allow probabilistic estimations of muzzle-to-target distance based on the overall elemental profile deposited on target materials. Such approaches have the potential to improve the reliability and discriminatory power of distance estimation by reducing ambiguity associated with individual elemental trends.

This research provides evidence that the relationship between muzzle-to-target distance and the concentration of inorganic gunshot residue deposited on the target material is non-linear and distance-dependent under controlled experimental conditions. The results demonstrate the feasibility of using quantitative elemental analysis as an investigative tool for shooting-distance assessment and establish a foundation for the future development of multivariate predictive models. Although additional validation across different firearms, ammunition types, target materials, and environmental conditions is required, the present study supports the potential use of quantitative IGSR profiling as a complementary analytical approach for forensic shooting-distance determination.

Data availability

Data will be available on request.

Competing interests

The authors declare no conflict of interest, particularly no financial and personal relationships with other people or organizations that could inappropriately influence (bias) this work.

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