# Shooting distance estimation based on gunshot residues analyzed by XRD and multivariate analysis

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**KEYWORDS**

Shooting distance; Multivariate analysis; X-ray diffraction

**ABSTRACT**

The most used and validated methods for estimating the shooting distance using the gunshot residues (GSR) in forensic labs are based on chemographic colour tests. In these techniques, the cloth-trapped residues are trans- ferred to a surface to be revealed using chemical reagents. However, because they imply a visual inspection, their interpretation may vary, thus adding possible errors to the forensic results. Therefore, it is important to find an objective analysis technique

for deciding during the results interpretation. In this study, X-Ray diffraction (XRD) was used to measure the GSR on cotton-polyester fabrics. The resulting diffractograms were aligned using a correlation optimized warping (COW) function, and then analysed using partial least squares to latent structures (PLS), and orthogonal PLS (OPLS). Both methods gave good prediction models in the 5–300 cm distance range, with determination coefficients of 0.99. Using the gun utilized during the shooting rendered good prediction models with quite small prediction errors (about 3 and 7%). Combining the two guns for the calculations, resulted in a prediction model with a larger prediction error (about 14%) but still good for predicting the shooting dis- tance. This would indicate that it is possible to use a similar gun to perform a shooting distance prediction without having the actual gun used during the investigated shooting.

## INTRODUCTION

Determining the shooting distance is crucial for how a casework may end at the courtroom. The shooting distance of a suicidal case normally differs from the distances attributed to other shootings like self-defence, burglary deterring, homicide, execution, etc. Each one of those crime scenarios (i.e., shooting distances) would receive a different penal treatment (sentence). The shooting distance is routinely inferred by analysing the amount and sort of certain specific material (i.e., gunshot residues, GSR) directly related to the ammunition used during the shooting [1,2]. GSR particles, depending on the type of gun or ammo, are normally left on the victim's body, the fired cartridge case, the barrel and chamber of the weapon, on the hands and body of the shooter, and other people in the firing place. The collection and identification of GSR allow associating a person with a

crime scene where a gun was shot. It also helps to estimate the shooting distance. GSR are microscopic and macroscopic particles resulting from the condensation of the vapour cloud produced by the high pressure, high temperature (ca. 2000–2500 ◦C) reactions that occur after a bullet is fired from a firearm.

Those normally spherical and spheroidal particles vary in size (ca. 0.1–5 μm), and are made up of material from the primer, bullet, bullet jacket, cartridge casing, and the gun barrel [3,4]. The European Network of Forensic Science Institutes Expert Working Group (ENFSI EWG) Firearms/GSR supports the chemographic-based methods [5,6], that use chemical reagents for visualizing particles con- taining a specific element or component that allow estimating the shooting distance. The most common colorimetric reagent is the sodium rhodizonate that reacts with Pb and Ba which are normally present in conventional ammo [7,8]. This technique has several inconveniences. Its calibration requires the use of the same gun, ammo at different distances. Besides, because this technique is colorimetric, it may render different results from one analyst to another according to their varying visual criteria. In addition, it is important to transfer the GSR to a device in order to visualize them, especially when studying dark clothes. Therefore, there may be a loss of precision due to the sample loss [9].

Several are the studies dealing with shooting distance using more advanced analytical techniques. Some of those use NAA [10], ICP-AES [11], and ICP-MS [12]. However, these techniques are destructive in nature. Nevertheless, other techniques are virtually non-destructive, like ATR-FTIR [13], commonly used in the distribution of organic residues, m-XRF [14], and VP-SEM-EDX [15], use for finding the distribution of the GSR inorganic elements. However, the analysis of particles

using these techniques require long times (several hours to days) [14].

XRD is the dominant technique in research. However, it has not been explored extensible like other non-destructive techniques or used for measuring shooting distances. In this case, the X-rays of known wave- length from an X-ray source, are used to probe the structure of the material, using the lattice plane of the sample as a diffraction grating. XRD is used for phase identification and distribution, to identify spatial arrangements of atoms in crystalline structure (phases), lattice strain fields, or stored defect content.

Previous analyses of non-jacketed projectiles (results not shown) [12] indicated that in many cases, Pb did not come from the primer but the bullet. In those cases, the analysed Pb had metallic properties, and it was the only metal detected in the diffractogram. Moreover, no traces (peaks) were found belonging to Sb, Ba, or other GRS-related element, even at the closest shooting distances. In order to detected them, their total con- centration has to be at least 1%, including the fabric's contribution.

To increase the identification rate of those heavy metals from the large amount of analysed spectroscopy data, some mathematical pro- cedures must be put in place in the analysis. Several chemometrics tools are broadly used for modelling many kind of biological and chemical data because they are efficient, validated, and robust methods [16]. Some of those tools are principal component analysis (PCA), linear discrimi- nant analysis (LDA), partial least squares to latent structures (PLS), orthogonal PLS (OPLS), and others.

In PLS, the found latent variables are linear combinations of the original variables. The criterion to select the weights is that the latent variable(s) describing the data matrix ($X$) should have maximal covariance with the data in the $y$ response matrix [17–19]. That is, the criterion is chosen for modelling $y$ as a function of $X$. When the $y$ data involves a single vector of data, the method is called PLS 1. When the problem relates two data tables to each other, the appropriate method would be PLS 2 [18].

OPLS, compared to PLS, rotates the model so that the correlated variation (related to the class separation) appears in the first predictive component, $tp$, while the uncorrelated variation (not related to the class separation) occurs in the orthogonal components [20,21]. While the prediction information offered by OPLS is basically the same as in PLS, the concepts of predictive and orthogonal components assist through the interpretation of the OPLS model.

This work focused on estimating the shooting distance using as parameter the metallic lead peaks obtained by X-ray diffraction from the GSR found on several experimental samples. Afterwards, the proposed method took the advantage of multivariate analysis, thus applied PLS on the cleaned spectra in order to calculate

valid models for predicting the shooting distance.

## MATERIALS AND METHODS

### Samples and standards

All the analyses were performed on GSR obtained after firing two different guns with CBC .38 ammunition (Companhia Brasileira de Car- tuchos, S-ao Paulo, Brazil) on various targets. This were provided and shot by ballistic experts at the Chilean Police. Fabrics were acquired in local markets of Santiago de Chile (Santiago de Chile, Chile).

Each target (evidence-like, test sample) was made with a 20 20 cm cardboard × base entirely covered with a piece of white cotton fabric. Subsequently, staff of the Ballistics section of the LACRIM Central (Policía de Investigaciones de Chile), made the shots on the fabrics. The shooting took place in the shooting room at the following distances: 5, 7.5, 10, 20, 30, 50, 100, 120, 150, 200, and 300 cm. Each shot was performed in triplicate with two guns of the same make: revolver Taurus calibre .38 special. After every shot, the target was collected and stored in Petri dishes. This was done to minimise the losses of GSR particles during the handling, and to avoid any contamination prior their XRD analysis.

## XRD CONDITIONS

At the analysis moment, the samples were cut into 6 6 cm squares around the× entrance hole of the bullet. This was done so the samples could be mounted on the diffraction sample holder. The equipment used was a XRD Empyrean (Malvern Panalytical, Malvern, UK). Table 1 shows the different parameters used for the

XRD measurements.

Before performing any calibration analysis on the data, the dif- fractograms were corrected using the correlation optimized warping (COW) function built-in the Unscrambler software (Camo, Oslo, Nor- way). This was done in order to align the peaks that otherwise would dramatically affect the data modelling.

The PLS and OPLS analyses were used to interpret to what extend the shooting distance can be correlated with the amount of GSR found on the shooting target. The multivariate analysis was performed with SIMCA (Sartorius Stedim Biotech, Aubagne, France). The data was previously centred and scaled (Unit Vector) which allowed eliminating any weight due to the variables or observations magnitude. Moreover, the software was set to calculate the boundaries with 95% Confidence level on the parameters, and 0.05 Significance level for the model distance and Hotelling's T2. The analysis was carried out using an automatic seven- groups' cross validation test.

## RESULTS AND DISCUSSION

For studying the origin of the metallic Pb and the capability of the technique to discriminate the shooting distance, every gun's set of results (triplicates) were analysed separately to isolate the factors affecting every gun.

**Table 1.** Parameters used for measuring XRD on the different samples.

| Parameter | Condition |
|---|---|
| Voltage | 45 kV |
| Current | 40 mA |
| Initial angle | 28° |
| Final angle | 90° |
| Slit width | 0,026 |
| Time through the slit | 159,375 s |
| Total time | 26:24 min |
| Incident beam | |
| Filter | Ni 0,02 mm |
| Soller Slit | 0,04 rad |
| Mask | 20 mm |
| Programmable divergent slit | 1/2° (0.75 mm) |
| Anti-scatter slit | 1° (1.50 mm) |
| Diffracted beam | |
| Soller Slit | None* |
| Programmable divergent slit | 2° (3 mm) |
| Sample | |

| | |
|---|---|
| movement | |
| Type of movement | Spinning |
| Revolution time | 4 s |

* It did not include a Soller slit because it incremented the signal in the detector.

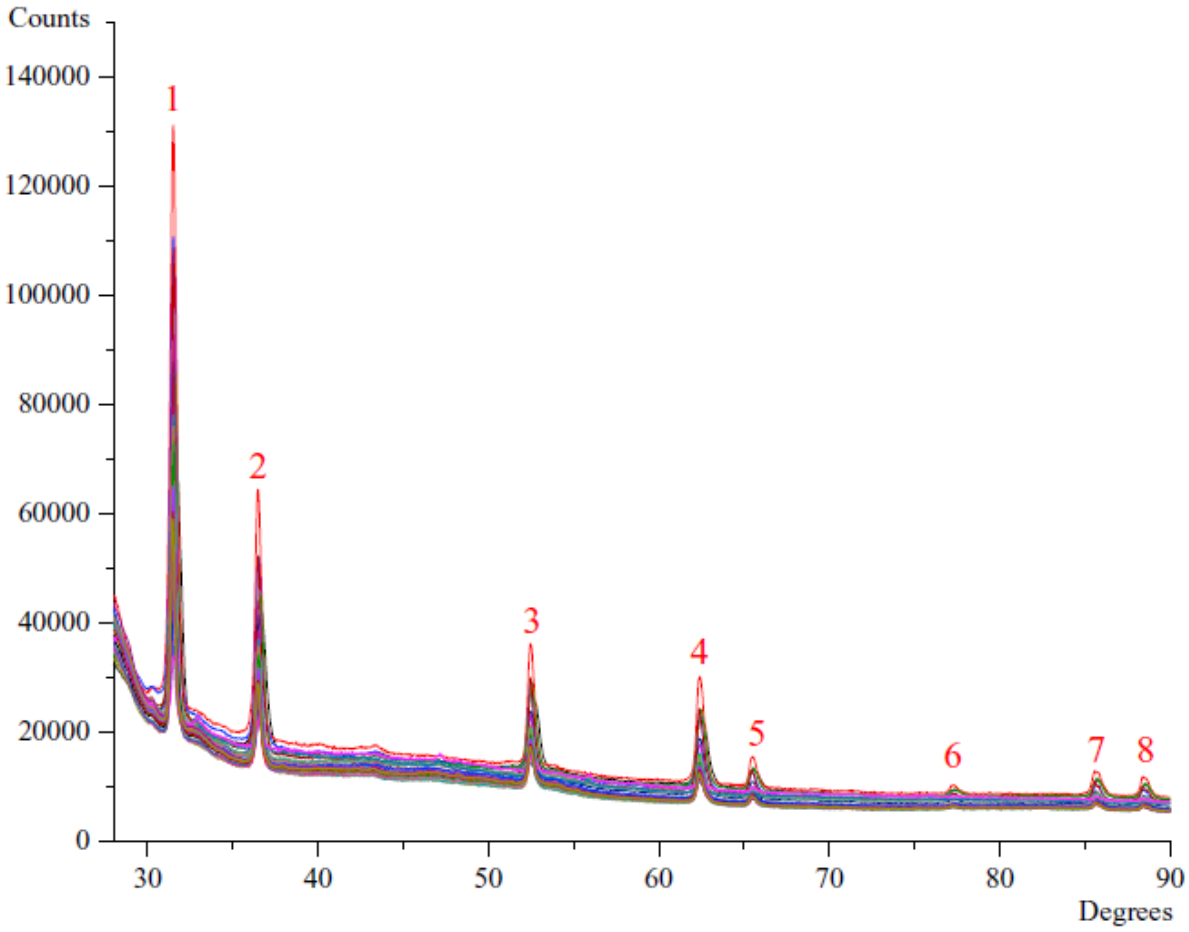


**Fig. 1.** X-ray diffractograms for the shooting distances between 28 y 90° on fabrics. The GSR particles were collected after firing a CBC 0.38 ammunition, and the curves were aligned using correlation optimized warping (COW).

Fig. 1 shows the X-ray diffractograms obtained for all distances. These diffractograms were compared against a database [22], showing that the predominant signals belonged to the cubic crystalline structure of Pb. In this case, Pb comes mainly from the projectile which agrees with previous studies [12]. They explained that the heat and hot gases produced during the propellent ignition act initially on the base of the projectile which have their metallic Pb exposed. The

first peak of the diffractograms represents the 100% intensity of the {1 1 1} plane with an inter-plane distance of 2.85 Å, the other peaks are described in Table 2. To check if it was possible to measure the shooting distance using the XRD technique, the GSR decay was studied. Considering the spectra (Fig. 1), it would be expected that the random noise in the baseline would weaken the models after autoscaling the data. That is, the autoscaling pre-treatment would put up a lot of weight on the random noise. Hence, it would be advised to perform a variable selection, and use just the eight well resolved lead peaks. However, using only those peaks became rather impractical (time consuming) and resulted in lower quality models. Therefore, in this study, the entire spectra region was used instead of just taking the peaks from lead. Fig. 2 shows that the shooting distance for the 5–300 cm range can be easily approximated to a potential relationship. This is in itself a very good result taking into account the long distances considered here. This approach used the peak at 50% intensity because the peak at 100% overlaps the fabric signal. This leads to a larger variation of the area which does not occur with the second peak that shows smaller variation.

**Table 2.** Parameters associated to the cubic crystalline structure of metallic Pb. The {h k l} values are the Miller indexes, d is the inter-plane distance, and I is the relative peak intensity.

| N° peak | h | k | l | d [Å] | I [%] |
|---|---|---|---|---|---|
| 1 | 1 | 1 | 1 | 2.85211 | 100.0 |
| 2 | 0 | 0 | 2 | 2.47000 | 50.3 |
| 3 | 0 | 2 | 2 | 1.74655 | 34.5 |

| | | | | | |
|---|---|---|---|---|---|
| 4 | 1 | 1 | 3 | 1.48947 | 39.9 |
| 5 | 2 | 2 | 2 | 1.42606 | 11.4 |
| 6 | 0 | 0 | 4 | 1.23500 | 5.2 |
| 7 | 1 | 3 | 3 | 1.13331 | 15.8 |
| 8 | 0 | 2 | 4 | 1.10462 | 14.7 |

From both multivariate methods tested in this work for each gun (PLS and OPLS), PLS performed better when using the entire 28–90° X-ray diffractograms for correlating the shooting distance and the amount of GSR found on the targets. The model for the first gun, for example, analysed 11 distances (5–300 cm) and was auto-fitted (according to the cross-validation criteria set in the software) for giving the minimum root mean square error of estimation (RMSEE). This was reached automati- cally after calculating eight latent variables (Fig. 3). Even so, less (and more) latent variables were manually estimated, however, they rendered worse models (larger RMSEE values), therefore, the auto-fitted latent variables were accepted. As it can be seen, this model was very good for estimating the shooting distance for the gun one. Fig. 3 shows how close the fitted distance values ($x$) are from the real shooting distances ($y$). This model had a RMSEE of about 7%.

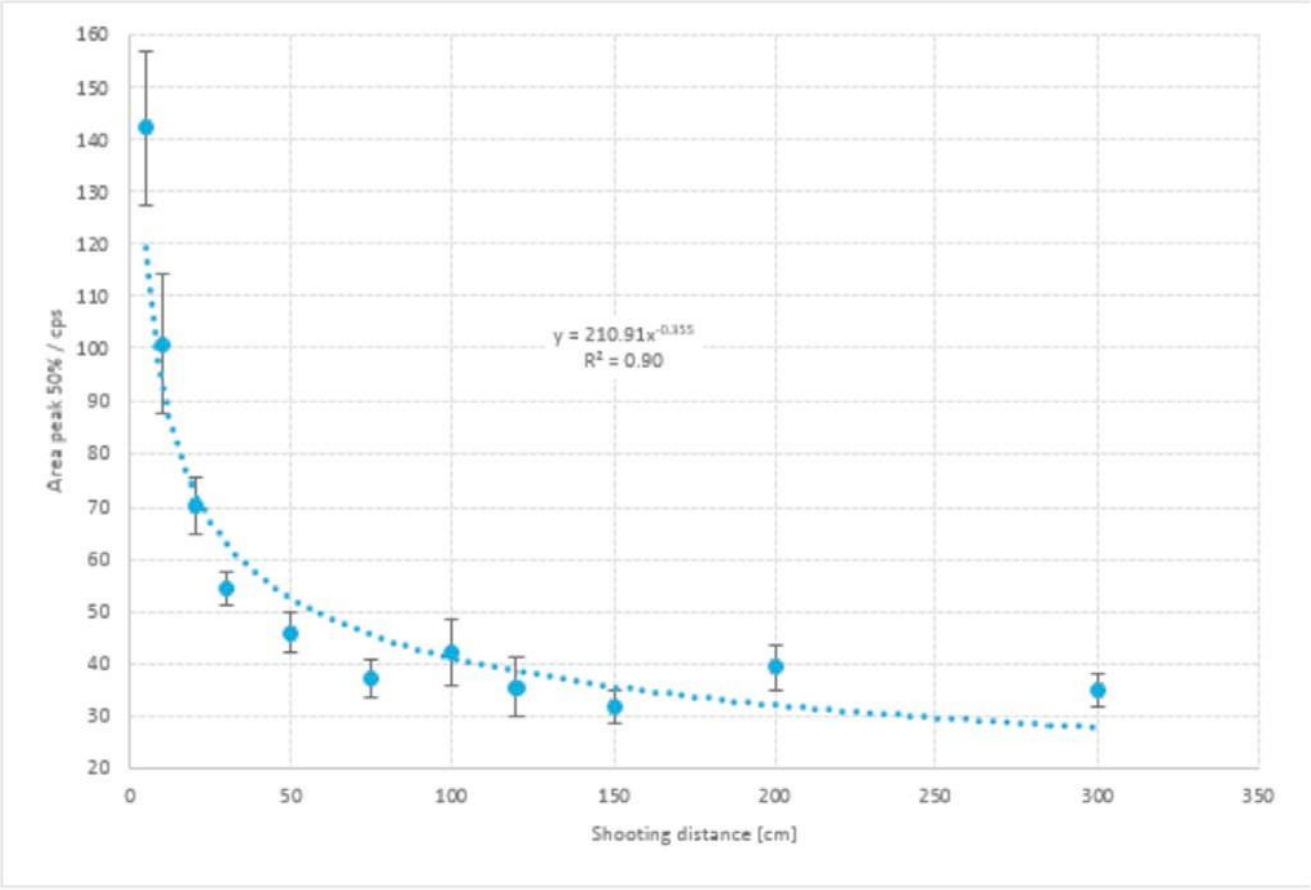


**Fig. 2.** Potential GSR decay between 5 and 300 cm. X represents the shooting distance while Y represents the area under the curve of the peak at the 50% intensity.

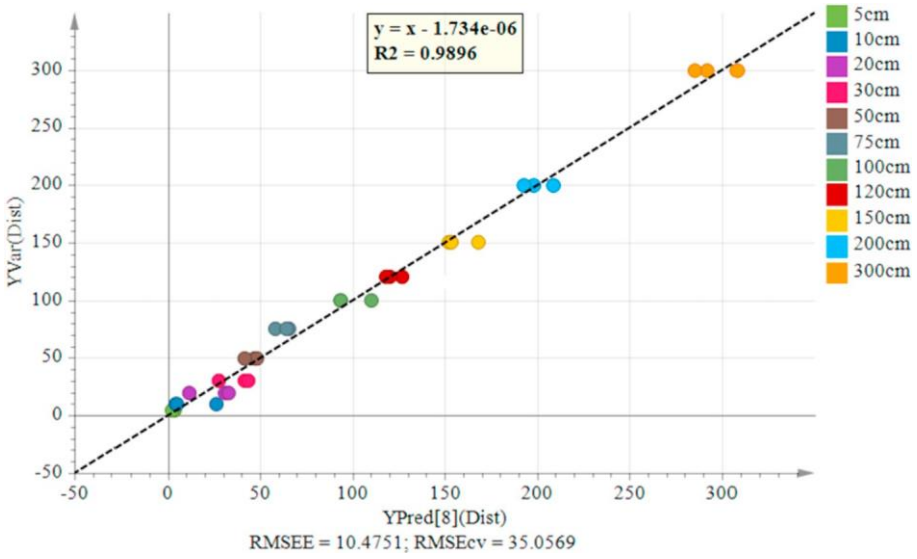


**Fig. 3.** PLS results for the observed versus predicted values of the Y-variable (Distance). The results belong to the data after shooting the gun one. X represents the predicted distances and Y represents the real shooting distances.

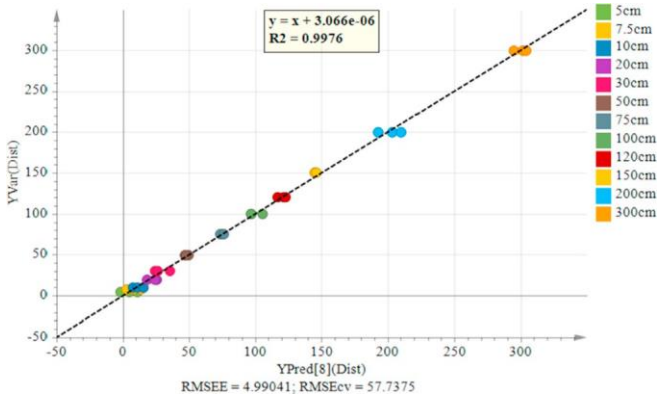


**Fig. 4.** PLS results for the observed versus predicted values of the Y-variable (Distance). The results belong to the data after shooting the gun 2. X represents the predicted distances and Y represents the real shooting distances.

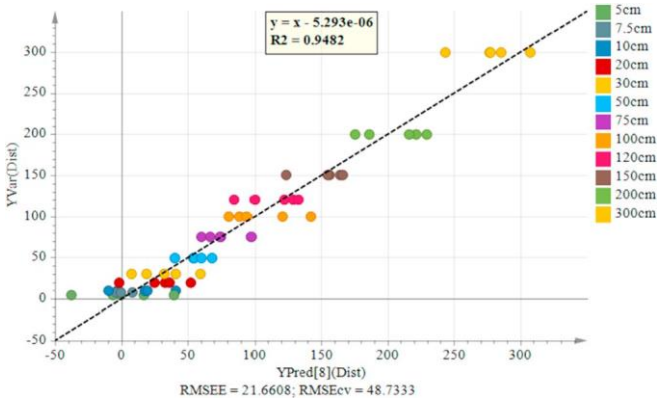


**Fig. 5.** PLS results for the observed versus predicted values of the Y-variable (Distance). The results belong to the data after shooting both guns. X represents the predicted distances and Y represents the real shooting distances.

Fig. 4 displays the PLS fitting results of the second model for the observed versus predicted values of the Distance *Y*-variable belonging to the gun two. Similarly, to the model one, this second model was also fitted with eight latent variables and proved to be very good for assessing the shooting distance for the gun two. This model had a RMSEE of about 3%

Fig. 5 displays the PLS fitting results of the third model describing the observed versus predicted values of the Distance *Y*-variable for the data of both guns together. This model, also fitted with eight latent variables, had less discrimination power but still was able to predict the shooting distance for both guns.

Despite that in forensic science it is well accepted the use of only one model per gun [10,23,24], in this case, combining both guns rendered a larger RMSEE (about 14%), which is still acceptable for a prediction model.

For this third model, an external validation set was used with samples comprising shooting distances between 50 and 300 cm, using both guns. This range of distances was chosen mainly because at lower distances the variation of the model is greater, which was observed for both the univariate and multivariate models. The prediction resukts for this third model are stated in Table 3, where an average error of 8% was obtained. These three models showed important results considering that the long distances are normally the most difficult to estimate given the scarce amount of GSR usually found on the scene.

Among the spectral pre-processing methods used in this study, only COW was the adequate for preparing the data whilst minimising its noise. This pre-treatment was necessary since the peaks in the diffractograms were misaligned. This frequent displacement occurs mainly when measuring directly on the fabric. Consequently, the GSR locate at dif- ferents heights, leading to a varied GSR granulometry, which in turn, produces shifts in the peaks.

Although the diffractograms were also treated with other type of pre-treatments, like base line and affset corrections, Savitzky-Golay smoothing, and standard normal variate normalization, none of them improved significantly the prediction models. What is worse, some pre- treatments like baseline and offset correction degraded the models completely. Therefore, only COW was performed as it rendered the best results.

Other studies used many test shots in order to increase the amount of GSR available for the analysis: 4–12 test shots from pure primers, 8–25 emitted from the barrel, 15–20 emitted from the breech [25]. Such procedure allowed them to identify some species like barium aluminate. Although the identification of the elements provided important clues regarding the residue origin and the physical and chemical processes that formed it. It is important to use a method enabling the identification and correlation of compounds and not only ions or elements. In that sense, this work presents a modern approach to use those renowned analytical techniques together with well-known chemometric methods in order to offer a quick tool for determining the shooting distance.

As expected, it is clear that using the same gun for predicting the shooting distance produced a better model with smaller prediction error compared to when the two guns were used. Nonetheless, the later model would still allow predicting the shooting distance. This is especially useful in cases where the shooting gun is missing or damaged, and thus no contrasting tests can be further performed.

**Table 3.** External validation of the PLS analysis using the distance model of both guns together.

| Gun | Real Distance | Predicted Distance | Error [%] |
|---|---|---|---|
| 1 | 50 | 41 | 18 |
| 2 | 75 | 69 | 8 |
| 1 | 100 | 92 | 8 |
| 2 | 120 | 118 | 2 |
| 1 | 150 | 131 | 13 |
| 2 | 200 | 200 | 0 |
| 1 | 300 | 270 | 10 |

## CONCLUSIONS

In the present work, despite that only one bullet was fired at each target, the combined analytical-multivariate techniques used showed a good accuracy for GSR particle detection and analysis. This was true regardless the gun used for the shooting. The correct identification of the particles allowed creating a good shooting distance calibration curve covering rather long distances.

XRD, compared to other techniques like SEM-EDX, used for detecting GSR, is less sensitive, letting detect only metallic Pb coming from the projectile. Nevertheless, this was not an obstacle and evidenced that this technique allowed creating a multivariate calibration model for predicting the shooting distance.

In this case, using the gun employed during the shooting resulted in good prediction models with rather small prediction errors of about 3 and 7%. Conversely, when the two guns were combined for the calculations, the prediction error increased significantly up to about 14%. Interestingly, this combined model could still predict well the shooting distance. This shows that it is possible to use a similar gun in order to perform a shooting distance prediction without having the actual gun used during the real shooting.

## NOVELTY STATEMENT

This manuscript shows the use of X-ray diffraction combined with multivariate analysis for predicting the shooting distance.

This study presents the proof of concept of a non-destructive, simple and fast approach that analyses gunshot residues (GSR) patterns in cotton-polyester-fabric targets shot with conventional caliber 0.38 Spe- cial ammunition.

The proposed novel prediction method achieved good results by harnessing the power of partial least squares to latent structure (PLS) and orthogonal PLS (OPLS).

## CRediT AUTHORSHIP CONTRIBUTION STATEMENT

Karla Leiva Miranda: Investigation, Formal analysis. Fernando E. Ortega-Ojeda: Formal analysis, Supervision, Validation, Writing - orig- inal draft, Writing - review & editing. Carmen García-Ruíz: Writing - review & editing, Visualization. Pedro Sáez Martínez: Conceptualiza- tion, Investigation, Resources, Supervision, Writing - review & editing.

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