

Lab Report XRF 431

S2 PICOFOX Forensic Distance Determination by TXRF after Firearm use



Introduction

Distance determination after firearm use is an important task during crime scene investigations. This requires trace element analysis of gunshot residue in the area surrounding the bullet hole. The major sources of gunshot residue are the ammunition's primer, which contain lead styphnate, barium nitrate and antimony sulfide compounds. However, some new primers do not contain lead, but can be characterized by other elements, e.g. Cu and Zn. In this report the determination of shooting distances for plumbiferous and unleaded ammunition by TXRF is described.

Instrumentation

All measurements were performed using the bench top TXRF spectrometer S2 PICOFOX. This instrument is equipped with an air-cooled low power X-ray tube (Mo target), a multilayer monochromator with 80% reflectivity and the liquid nitrogen-free XFlash® Silicon Drift Detector (SDD) with an energy resolution of < 159 eV (Mn K α).

Experiments and sample preparation

Shooting experiments were performed on white scrim according to the parameters in table 1. After shooting of four textile samples, an area with a diameter of 46 mm around the bullet hole was cut out. The large area was required for simultaneous wavelength-dispersive XRF analysis.

The textile samples were treated with 2 ml aqua regia for two hours at room temperature. Subsequently, the samples were filled up with 38 ml distilled water according to the standard procedures for atomic spectroscopy or ICP-MS analysis. TXRF allows the analysis of undiluted samples.

Table 1: Parameters for the shooting experiments

Ammunition	Ammunition types	Shooting distances (cm)
GECO 7.65 mm, Browning	plumbiferous	20, 50, 80, 110, 150
Sintox Fiocchi, 9 mm, Luger	lead-free	20, 40, 60, 80

Table 2: TXRF results for the gunshot residue elements

Shooting distance (cm)	Plumbiferous ammunition	
	Ba ($\mu\text{g/l}$)	Pb ($\mu\text{g/l}$)
20	476 +/- 65	950 +/- 80,3
50	77,6 +/- 21,7	78,4 +/- 16,7
80	18,7 +/- 3,7	9,1 +/- 0,56
110	16,4 +/- 14,0	3,4 +/- 0,27
150	12,4 +/- 0,46	2,8 +/- 0,56

Shooting distance (cm)	Lead-free ammunition	
	Cu ($\mu\text{g/l}$)	Ba ($\mu\text{g/l}$)
20	623 +/- 155	683 +/- 165
40	111 +/- 14,5	200 +/- 22,5
60	40,8 +/- 10,1	87,8 +/- 22,6
80	12,8 +/- 1,6	66,4 +/- 6,7

Aliquots of 1 ml solution were taken from each sample for TXRF analysis. For internal standardization of the unleaded samples 10 μl of an Y solution (Merck, 100 mg/l) and in case of plumbiferous samples 10 μl of a Se solution (Merck, 100 mg/l) were added, respectively. After thorough homogeniza-

tion 10 µl of each sample were transferred to quartz glass sample carriers and dried in vacuum. Measurement time for all samples was 1000 s.

The results for the gunshot residue elements are listed in table 2. Sb was not analyzed due to the interference of the L-lines of Sb with the K-lines of the abundant element Ca. The concentrations of Ba and Pb are showing a distinct correlation to the applied shooting distance (figure 1), which allows the determination of shooting distances for plumbiferous ammunition up to a distance of 110 cm.

The correlation of the element concentrations and the shooting distance for unleaded ammunition is shown in figure 2. Zn is ubiquitarily present and for that reason cannot be used for shooting distance determinations. In contrast, the data for Ba and Cu show good correlation and are suitable for the determination of even larger distances.

Conclusion

The results of the presented measurements clearly indicate the suitability of the TXRF spectrometer S2 PICOFOX for distance determination after firearm shooting. In contrast to other analytical methods like atomic spectroscopy, no external calibration is necessary. The simultaneous determination of all detectable elements by TXRF is possible also in case of unknown ammunition or element concentrations. Therefore, TXRF offers the flexibility to handle future changes of primer compositions.

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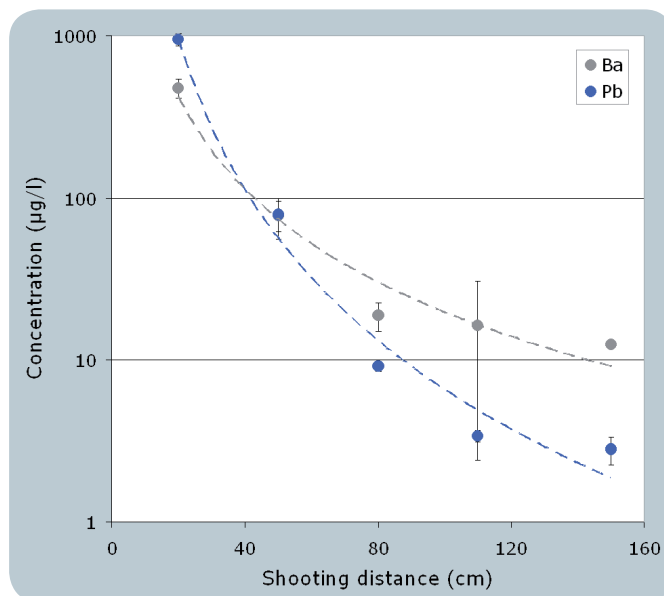


Figure 1: Correlation of gunshot residue concentrations with the shooting distance for plumbiferous ammunition

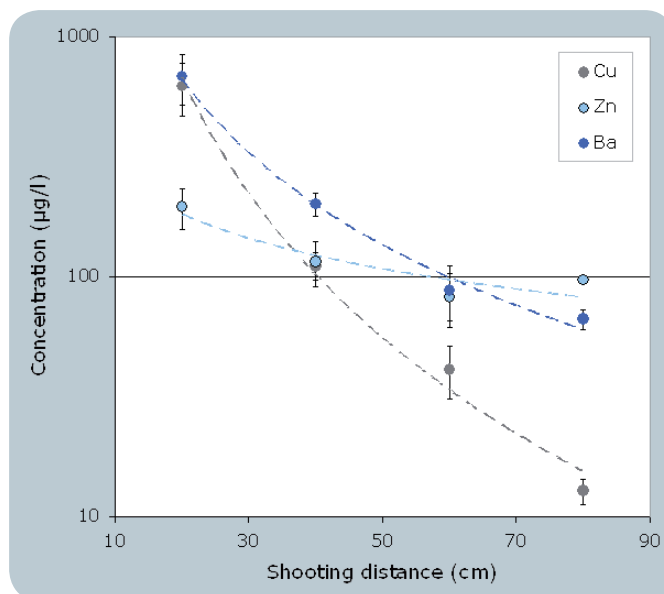


Figure 2: Correlation of gunshot residue concentrations with the shooting distance for lead-free ammunition

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