



### A109 Analysis of Lead-Free Ammunition by Scanning Electron Microscopy Using Energy Dispersive X-Ray Spectroscopy and Discrimination of Samples Using Multivariate Statistical Methods

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The goal of this presentation is to explore statistical methods for differentiating non-traditional Gunshot Residue (GSR) samples arising from six non-toxic, lead-free ammunitions. The formulation of non-toxic ammunition excludes the traditional signature composition of GSR, a mixture of lead (Pb), barium (Ba), and antimony (Sb). The removal of the unique combination of these three elements also renders differentiation of non-toxic GSR from environmental sources more challenging.

This presentation will impact the forensic science community by further exploring the combinatorial feasibility of Energy Dispersive X-ray Spectroscopy (EDS) and multivariate statistical analysis through the characterization of relatively understudied non-toxic ammunition. The application of these procedures allows for the statistical comparison of spectra, rather than the potentially more subjective visual comparisons. In turn, the statistical procedures allow comparisons to be made based on mathematical principles with measureable confidence in the comparison.

Traditionally, during the identification of GSR both morphological features of particles and elemental compositions are determined. In most laboratories, the presence of spherical particles containing Pb, Ba, and Sb indicates the definitive presence of GSR. However, the emergence of lead-free ammunition in response to health and environmental concerns requires a broader definition of particle compositions consistent with GSR. Unlike conventional primers, manufacturers of non-toxic ammunition may use vastly different elemental compositions to achieve the final product. For example, aluminum, silicon, potassium, strontium, and calcium may be included in the primer. Thus, no single particle composition can be easily amended to the description of GSR.

This study collected elemental profiles for six commonly available brands of non-toxic ammunition as well as for the most common brand of road safety flare utilized in the United States. Flares were included in the study due to their high strontium content and their potential for similarity in element composition to non-toxic ammunition. For each brand, one box of 9mm caliber ammunition was purchased and five rounds from each box were fired in the presence of three conductive carbon tabs positioned along the trajectory of the bullet, allowing capture of GSR. An additional five rounds from each box were disassembled to remove the propellant and bullet before being fired. These rounds were fired perpendicular to two conductive carbon tabs, allowing for the capture of only primer components. Road flares were burned in the presence of two conductive tabs positioned above the flare to capture any ejected particles during the combustion process.

A carbon tab was analyzed from each of the resulting 61 samples using a Scanning Electron Microscope (SEM). Particles visually consistent with the morphology of GSR were located on the tabs at an accelerating voltage of 20 keV. Once potential particles were discovered, the visual area of the sample was designated as a region of interest and all particles within view were analyzed by EDS. Searching of each sample finished after 30 regions of interest were identified. Each EDS spectrum was analyzed from 0-10 keV for elemental composition determination. Several different data pretreatment methods (including scaling and normalization procedures) were applied to the resulting spectra prior to statistical analysis. The pretreated data were then analyzed using Principal Components Analysis (PCA). This procedure is a useful visualization tool where samples with similar EDS spectra cluster closely in the resulting scores plot while samples with different spectra position further apart. In this study, PCA was first used to investigate association of ammunition by manufacturer, based on the element profiles generated. Then, the association of samples from whole bullets to those from primers only, and the differentiation of actual GSR from a ubiquitous road flare, were also investigated using PCA.

The application of data pretreatment and statistical procedures for SEM-EDS examinations of GSR allows greater confidence in results from a technique that is traditionally thought of as merely qualitative. Likewise, the lessons learned from the EDS analysis of GSR may also be applicable in the comparison of other items of trace evidence based on element composition, for example, comparisons of glass fragments or paint samples.

**Gunshot Residue, SEM-EDS, Chemometrics**