A153 Metrological Issues Concerning the Analysis of Brazilian Heavy-Metal Free GSR by SEM

Andrea Martiny, PhD, Av Nossa Senhora das Graças, 50 Bldg 27, Xerem, Duque de Caxias, BRAZIL; and Andrea P. Campos, PhD, Av Nossa Senhora das Graças, 50 Bldg 3, Xerém, Duque de Caxias, BRAZIL*

After attending this presentation, attendees will understand issues regarding the size and composition of Gunshot Residue (GSR) particles that may affect and even impair an accurate result.

This presentation will impact the forensic science community by discussing unique metrological issues that are usually overlooked during GSR analysis, recognizing the differences from the pressure to produce environmental ammunitions may impair the accurate identification of GSR by all known methodologies.

Metrology is defined as the science that includes all theoretical and practical aspects of measurement. Although not disseminated among the forensic science and justice communities, it is of utter importance since accurate measurement results can have an impact on the outcome of a trial. Most forensic analysis relies on qualitative techniques, which probably explains why metrology is so overlooked. Yet even qualitative analysis can benefit from metrology. One such example is GSR particle analysis. GSR is probably the most common and at the same time underestimated type of trace evidence, despite the growing number of firearms-related offences in Brazil. GSR particles are generated by a combination of high temperature and high pressure conditions occurring during firearm discharge. Particles are mainly composed of elements from primer mixture (mostly heavy metals) and so are very stable. They are also non-crystalline and circular in shape. The technique of choice is SEM-EDX and results are expressed in qualitative terms: presence or absence of particle types as defined by the ASTM E-1588/10e1 standard. However, the detection of particles and its identification by automated SEM systems is greatly dependent on metrology. For instance, identification is based on particle diameter range and element composition. In this study, metrological aspects of GSR detection were analyzed by SEM. Automated GSR particle analyzer software base their identification on three parameters: BSE brightness given by Z contrast, circularity factor, and element composition. These parameters rely on the ability of the instrument (i.e., the SEM and the EDX) to measure with accuracy. This implies that the BSE detector and X-ray probe must be adjusted and magnification calibrated.

Validation of the method was performed employing Ted Pella SPS-5P-2 GSR Certified Standard, ENFSI synthetic GSR standard, and an in-house GSR-like sample containing LaCe(Fe) particles (0.5-20µm). In the case of this research instrument, an FEI Quanta 200 ESEM equipped with a Genesis EDAX EDX and GSR-XT software, the instrument detection limit (IDL = 0,8) was calculated in regard to the smallest particle size detected, employing the CRMs, meaning that smaller particles were not detected, except in manual search. The sub-micron detection capability of laboratories from four different proficiency tests using a similar RM were reviewed and found that approximately 50% were considered unsatisfactory for 0.5µm particles. This percentage is steady from 2001 to 2008. These laboratories are recommended to disregard sub-micron GSR during real examinations. Yet, the inability to detect sub-micron particles is critical for Brazilian samples, due to the tendency of CBC primer to generate such smaller particles. Accuracy was tested applying the Z-Score to the results of seven runs and was considered unsatisfactory (the mean z was -9,98, meaning that results were almost 10x below the correct mean number) when the software was installed. At this time, on average 90% of the 2.4µm particles were detected. Satisfactory mean Z-scores were achieved after calibration of magnification using a CRM and of X-ray probe adjust employing a metal CRM, resulting in the detection on average of 90% of all 0.8µm particles. Although a rather acceptable reproducibility was obtained (same sample and parameters over different days and operators), repeatability (same sample and parameters over a short period of time) was not acceptable (*s.d.* = ± 3) against a preferred 1.3 for 0.5µm particles. A great concern resulting from the analysis of Brazilian ammunition generated-GSR is the size of particles. GSR range between 0.5 and 20µm in diameter, but particles generated by CBC ammunition are mostly sub-micron. On one hand, this may increase its potential as a hazardous airborne particle, although it also imposes some difficulties in detection by most SEM systems because of: (1) pixel limitations; and, (2) electron transparency. A minimum 20kV accelerating voltage is required to detect PbL lines, but this may be a problem for sub-µm particles because of the interaction volume and reduced number of BSE. Most systems detect particles >0.5µm, which in reality corresponds to 0.7µm particles. The characteristics arising from CBC-generated GSR probably mean that a smaller beam diameter is needed and associated to a rigorous control of SEM parameters to ensure accurate results.

This research was supported by FINEP grant # Metrofor 01.10.0715.00 and CNPq/Prometro Grant # 563093/2010-2.

Gunshot Residues, Microscopy, Metrology