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Standard Guide for Gunshot Residue Analysis by Scanning Electron Microscopy/ Energy Dispersive X-ray Spectrometry[1]

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1. Scope

1.1 This guide covers the analysis of gunshot residue (GSR) by scanning electron microscopy/energy-dispersive X-ray spectrometry (SEM/EDS) by manual and automated methods. The analysis may be performed manually, with the operator manipulating the microscope controls and the EDS system software, or in an automated fashion, where some amount of the analysis is controlled by pre-set software functions.

1.2 Since software and hardware formats vary among commercial systems, guidelines will be offered in the most general terms possible. The software manual for each system should be consulted for proper terminology and operation.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Practice

2.1 From the total population of particles collected, those that are determined by SEM to be within the limits of certain parameters (for example, atomic number, size, or shape) characteristic of or consistent with GSR are analyzed by EDS. Typically, particles composed of high mean atomic number elements are detected by their SEM backscattered electron signals and an EDS spectrum is obtained from each. The EDS elemental profile is evaluated for constituent elements that may identify the particle as being characteristic of or consistent with GSR.

3. Significance and Use

3.1 This document will be of use to forensic laboratory personnel who are involved in the analysis of GSR samples by SEM/EDS.

3.2 SEM/EDS analysis of GSR is a non-destructive method that provides[2,3] both morphological information and the elemental profiles of individual particles. This contrasts with bulk sample methods, such as atomic absorption spectrophotometry, neutron activation analysis, inductively coupled plasma atomic emission spectrometry, and inductively coupled plasma mass spectrometry, where the sampled material is dissolved or extracted prior to the determination of total element concentrations, thereby sacrificing morphological information and individual particle identification. In addition, X-ray fluorescence spectrometry (XRF) is a bulk analysis technique that has been used for the elemental analysis of GSR. Unlike the solution-based bulk methods of analysis, XRF is nondestructive; however, XRF still does not provide morphological information and is incapable of individual GSR particle identification.

4. Sample Preparation

4.1 Once the evidence seal is broken, care should be taken so that no object touches the surface of the adhesive SEM/EDS sample collection stub and that the stub is not left uncovered any longer than is reasonable for transfer, mounting, or labeling.

4.2 Label the sample collection stub in such a manner that it is distinguishable from other sample collection stubs without compromising the sample; that is, label the bottom or side of the stub.

4.3 If a non-conductive adhesive was used in the sample collection stub, the sample will need to be coated to increase its electrical conductivity, unless an environmental SEM or low pressure/low vacuum - SEM is used for the analysis. Carbon is a common choice of coating material, since it will not be detected with a beryllium window EDS detector and, thus, will not interfere with X-ray lines of interest. Furthermore, with EDS systems capable of detecting carbon, it is still ignored due to the high signal intensity from the carbon in the adhesive. For high vacuum SEM, a carbon film thickness of between 5 and 50 nm is typical, with less conductive samples requiring a thicker coat. If the carbon coating thickness is not measured,

[1] This guide is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.01 on Criminalistics.

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[2] Krishnan, S. S., "Detection of Gunshot Residue: Present Status," *Forensic Science Handbook*, Volume I, Prentice Hall, Inc., Englewood Cliffs, NJ, 1982.

[3] Wolten, G. M., Nesbitt, R. S., Calloway, A. R., Loper, G. L., and Jones, P. F., "Final Report on Particle Analysis for Gunshot Residue Detection," *Report ATR-77 (7915)-3*, Aerospace Corporation, Segundo, CA, 1977.

then its effectiveness in reducing sample charging to an acceptable level should be confirmed prior to analyzing evidentiary samples.

5. Sample Area

5.1 Sample collection stubs for SEMs typically come in one of two diameters: 12.7 mm (0.5 in.) or 25.4 mm (1 in.), which yield surface areas of 126.7 mm^2 and 506.7 mm^2 respectively. Analysis of the total surface area of the stub manually is prohibitively time-consuming. Because the particles are collected onto an adhesive surface in a random manner and the particles do not tend to cluster, it is reasonable to analyze a portion of the stub surface by employing an appropriate sampling and analytical protocol.[3,4]

5.2 When an automated SEM/EDS system is employed, data collection from the entire surface area of the sample collection stub is recommended if possible. Due to the disparity between the shape of the sample collection stub (round) and the SEM field of view search area (square or rectangular), analysis of 100% of the sample collection area may not be possible in some systems.

6. Instrument Requirements and Operation

6.1 *General*:

6.1.1 Most commercial-grade SEM/EDS systems should be adequate for GSR analysis.

6.1.2 Automated data collection of GSR involves some portion of the data collection being controlled by pre-set software functions. The extent to which the SEM and EDS systems communicate and are integrated varies according to the manufacturers involved and the capabilities of the hardware/software architecture.

6.1.3 A protocol should be established to confirm optimal operating parameters on a routine basis.

6.1.3.1 The EDS energy calibration and beam current stability should be monitored regularly.

6.1.4 If a reference sample with a known amount of particles (preferably GSR particles) is available, this sample (positive control) should be analyzed in regular intervals in order to test the accuracy of particle detection, whether by automated or manual analysis.[5]

6.1.5 A stub that has not been used for collection (negative control) should also be included with each sample set analyzed.

6.2 *Scanning Electron Microscope (SEM)*:

6.2.1 The SEM, operating in the backscattered electron imaging mode, must be capable of detecting particles down to at least 0.5 µm in diameter.

6.2.2 The SEM must be capable of an accelerating voltage of at least 20 kV.

6.2.3 Automated systems will also include:

6.2.3.1 a motorized stage

6.2.3.2 automated stage control with the ability to recall stage locations of particles for verification

6.2.3.3 particle recognition software

6.3 *Energy Dispersive Spectrometry (EDS)*:

6.3.1 *Detector:*

6.3.1.1 The detector window may be constructed either of beryllium or organic "thin" film[6].

6.3.1.2 The detector's resolution should be better (less) than 150 eV, measured as the full width at half the maximum height of the Mn Kα peak.

6.3.1.3 The detector must be capable of resolving clearly the Ba $L\alpha_1$, $L\beta_1$, and $L\beta_2$ peaks.

6.3.2 *Display:*

6.3.2.1 A calibrated, scaled display of X-ray energy versus counts.

6.3.2.2 The ability to identify and label X-ray lines and a facility for hard copy output of the display contents.

6.3.2.3 At a minimum, the display should be set to 1024 channels at 20 eV per channel. If the software allows, the EDS display should be set to 2048 channels at 10 eV per channel as this permits better visualization (resolution) of the X-ray lines.

6.3.2.4 Display of the EDS output must encompass the X-ray lines of analytical utility, with a minimum range of 0-15 keV.

6.3.3 Automated systems will also include:

6.3.3.1 software capable of acquiring for a specified collection time or total counts and storing EDS spectra from multiple points on the sample collection stub.

6.4 *Sample Placement*:

6.4.1 For identification purposes, each sample collection stub should contain a permanent or indelibly labeled identifier which can be used to reference the stub location on the microscope stage.

6.4.2 If it is anticipated or required that additional analyses or particle relocation will occur after a stub has been analyzed and removed from the microscope stage, a system should be devised so the stub can be replaced in the same orientation as before its removal. This may consist of marking the side of each stub and aligning it with marks on the microscope stage or by having stubs that fit into the stage in only one position (for example, stubs with a pin that is a half-circle in cross-section).

6.5 *Detection and Calibration*:

6.5.1 Particles of GSR are detected by their backscattered electron signal intensity. The absolute signal intensity that a particle produces is related to the electron beam current, mean atomic number, and size of the particle (for particle sizes on the order of the beam diameter). Particles whose mean atomic numbers are high will appear brighter than those of lower mean atomic number composition. As the beam current increases, the amount of signal each particle produces also increases.[7]

[4] Halberstam, R. C., "A Simplified Probability Equation for Gunshot Primer Residue (GSR) Detection," *Journal of Forensic Sciences*, V36, N3, pp. 894–897, 1991.

[5] A reference sample should have been prepared and mounted in a manner comparable to the collection method in use by the submitting agency. Preferably, the calibration sample will be a sample of GSR from a known source (caliber of weapon, ammunition manufacturer, number of rounds fired, collected area from shooter or a synthetic GSR standard. Additional environmental particles may be added to ensure the inclusion or exclusion of particular classes of particles.

[6] A beryllium window absorbs X-ray energies below about 1.0 keV; therefore, elements below sodium (atomic number 11) are not detected; other window materials may be employed (single window light element detectors).

[7] Goldstein, J. I., Newbury, D.E., Echlin, P., Joy, D.C., Lyman, C.E., Lifshin, E., Sawyer, L., and Michael, J.R.; Scanning Electron Microscopy and X-Ray Microanalysis, 3rd Edition, Kluwer Academic/Plenum Publishers, 2003.