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Standard Guide for Gunshot Residue Analysis by Scanning Electron Microscopy/ Energy—Dispersive Spectroscopy[1]

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1. Scope

1.1 This guide covers the analysis of gunshot residue (GSR) by scanning electron microscopy/energy-dispersive spectrometry (SEM/EDS) by manual and automated methods. The analysis may be performed "manually," with the operator manipulating the microscope stage controls and the EDS system software, or in an automated fashion, where some amount of the analysis is controlled by pre-set software functions requiring little or no operator intervention.

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

2. Referenced Documents

2.1 *ASTM Standard:*

E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data[2]

3. Summary of Practice

3.1 Particles composed of high mean atomic number elements are detected by their backscattered electron signals and an EDS spectrum is obtained from each. The elemental profile is evaluated for constituent elements which may identify the particle as being unique to or indicative of GSR.

4. Significance and Use

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

4.2 Analysis of GSR by SEM/EDS currently provides a highly definitive method of identification,[3,4] because it assigns an elemental profile to a particular particle. This contrasts with a bulk sample method, such as atomic absorption or neutron activation analysis, where the total measured levels of the elements in question are not distinguishable from non-GSR sources.[3,4]

5. Sample Preparation

5.1 Once the evidence seal is broken, care should be taken so that no object touches the surface of the collection stub and that the specimen stub is not left uncovered any longer than is reasonable for transfer or mounting.

5.2 Label the stub in such a manner that it is distinguishable from other specimen stubs without compromising the sample's analysis, that is, label the bottom or side of the stub.

5.3 If a non-conductive adhesive was used in the collection device, the sample will need to be coated to increase its electrical conductivity. Carbon is a common choice of evaporant, since it will not be detected with a beryllium window EDS detector and, thus, will not interfere with X-ray lines of interest. A thickness of between 5 and 50 nm is typical, with more non-conductive samples requiring a thicker coat.

6. Sample Area

6.1 Sample stubs for SEMs typically come in one of two diameters: 12.7 mm (0.5 in.) or 25.4 mm (1 in.); these yield surface areas of, respectively, 126.7 mm^2 and 506.7 mm^2. To manually analyze the total surface area of the stub is prohibitively time-consuming; since the distribution of particles collected onto an adhesive surface is random and the particles do not tend to cluster,[4,5] appropriate sampling regimes may be employed.

6.2 This relationship may also be expressed in terms of the portion of the area that must be searched to ensure the finding, with some arbitrary certainty, of at least one GSR particle, based on a predetermined population on a stub:

$$p = 1 - p^{1/N} \quad (1)$$

Thus, for example, if a jurisdiction required the identification of a minimum of 5 GSR particles on a stub for a positive

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[2] *Annual Book of ASTM Standards*, Vol 03.06.

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

[4] 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.

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