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~~Designation:E1588–08~~ Designation: E1588 – 10

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

This standard is issued under the fixed designation E1588; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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.

~~1.2Since 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.~~

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1.2 Since software and hardware formats vary among commercial systems, guidelines will be offered in the most general terms possible. For proper terminology and operation, consult the SEM/EDS system manuals for each system.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.*

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2. Summary of Practice

2.1 From the total population of particles collected, those that are ~~determined~~detected 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 (**1-3**).[2] 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~~ spectrum is evaluated for constituent elements that may identify the particle as being ~~characteristic of~~consistent with or ~~consistent with GSR.~~ characteristic of GSR, or both.

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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 (**4**).

3.2 SEM/EDS analysis of GSR is a non-destructive method that provides (**5, 6**) both morphological information and the elemental profiles of individual particles. ~~This~~

3.3 Particle analysis contrasts with bulk sample methods, such as atomic absorption spectrophotometry (AAS) (**7**), neutron activation analysis (NAA) (**8**), inductively coupled plasma atomic emission spectrometry (ICP-AES), and inductively coupled plasma mass spectrometry (ICP-MS), 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~~

3.4 X-ray fluorescence spectrometry (XRF) is a ~~bulk analysis~~ technique that has been used ~~for~~ to map the ~~elemental analysis~~ placement and distribution of GSR particles surrounding bullet holes in order to establish shooting distances (**9**). Unlike the solution-based bulk methods of analysis, XRF is non-destructive; 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.

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[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.

~~Current edition approved March 15, 2008. Published April 2008. Originally approved in 1994. Last previous version approved in 2007 as E1588–07e1. DOI: 10.1520/E1588-08.~~

Current edition approved June 1, 2010. Published June 2010. Originally approved in 1994. Last previous version approved in 2008 as E1588 – 08. DOI: 10.1520/E1588-10.

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

[2] The boldface numbers in parentheses refer to a list of references at the end of this standard.

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~~4.2Label 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.~~

~~4.3If 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, then its effectiveness in reducing sample charging to an acceptable level should be confirmed prior to analyzing evidentiary samples.~~

4.2 Label the sample collection stub in such a manner that it is distinguishable from other sample collection stubs without compromising the sample; for example, 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 variable-pressure/low-vacuum SEM is used for the analysis. Carbon is a common choice of coating material, since it will not interfere with X-ray lines of interest. For high-vacuum SEM, coat the sample sufficiently to eliminate charging of the sample.

5. Sample Area

~~5.1Sample 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~~

5.1 Sample collection stubs for SEMs typically come in one of two diameters: 12.7 mm or 25.4 mm, 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]~~

~~5.2When 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.~~ respectively.

5.2 Manual analysis of the total surface area of the stub 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 **(6, 10)**.

5.3 Automated SEM/EDS analysis can enable data collection from nearly the entire surface area of the sample collection stub. 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.

5.3.1 Analysis of the maximum allowable surface area of the sample is recommended, however, many automated systems can be programmed to terminate the analysis of a stub or series of stubs once a pre-established number of particles with specified classification(s) have been detected. The decision as to how many particles satisfy the requirements of a particular case is a matter for the analyst to decide but should be subject to guidelines set out in the laboratory's standard operating procedures.

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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.

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

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

~~6.1.4If 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.~~

~~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)*:

~~6.2.1The 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.~~

~~6.2.3Automated systems will also include:~~

~~6.2.3.1a motorized stage~~

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

~~6.2.3.3particle recognition software~~

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 SEM/EDS systems include: communication and control between the SEM and EDS system, and a motorized stage with automated stage control. The system should have the ability to recall stage locations of particles for verification and software for particle recognition.

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.~~

~~6.3.1.2The 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.~~

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

~~6.3.2*Display:*~~

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

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

~~6.3.2.3At 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:~~

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

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

6.3.2 At a minimum, the EDS spectrum should be acquired at 20 eV per channel.

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

6.3.4 Automated systems will also include software capable of acquiring X-ray spectra for a specified collection time or total X-ray counts.

6.3.5 It is desirable that the spectrum obtained from the analysis of each particle of interest be stored. At a minimum, an automated system must be capable of storing all of the particle location coordinates.

6.4 *Sample Placement*:

~~6.4.1For 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.~~

~~6.4.2If 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).~~

6.4.1 Record the positions of the stubs (sample and standard/reference stubs) on the SEM stage when the samples are inserted.

6.4.2 If it is anticipated or required that additional analyses will be needed, it is desirable that the stub can be returned to 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*:

~~6.5.1Particles 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.~~

~~6.5.2The brightness and contrast settings of the backscattered electron detector system determine the limits of detection and discrimination of particles whose mean atomic number exceed the minimum setting but fall below the maximum setting. Controls for the backscattered electron signal should be set on a suitable reference sample of known origin or a pure element standard at the same parameters that will be used for the questioned sample analysis. This calibration sample should, if possible, be in the microscope chamber at the same time as the questioned samples to be analyzed.~~

~~6.5.3The backscattered electron detector's brightness and contrast should be set to include those high atomic number particles of interest and exclude low atomic number particles that are not of interest. Typically, high contrast and low brightness settings provide an adequate range between thresholds for ease of detection. If the beam current is changed or drifts, the brightness and contrast, which were based on the previous beam current, may no longer be compatible with the new conditions and should be readjusted. The beam current may be measured with a Faraday cup, a specimen current meter, or monitored by comparing the integrated counts within the same peak in sequentially collected spectra from a known standard.~~

6.5.1 Particles of GSR are detected by their backscattered electron signal intensity. The absolute signal intensity that a particle