



# Standard Practice for Gunshot Residue Analysis by Scanning Electron Microscopy/Energy Dispersive X-Ray Spectrometry<sup>1</sup>

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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice covers the analysis of gunshot residue (GSR) by scanning electron microscopy/energy-dispersive X-ray spectrometry (SEM/EDS). The analysis is performed using automated software control of both the SEM and EDS systems, to screen the sample for candidate particles that could be associated with GSR. Manual control of the instrument is then used to perform confirmatory analysis and classification of the candidate particles. This practice refers solely to the analysis of electron microscopy stubs (1).<sup>2</sup>

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 instrument.

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 cannot replace knowledge, skills, or abilities acquired through education, training, and experience (Practice E2917), and is to be used in conjunction with professional judgment by individuals with such discipline-specific knowledge, skills, and abilities.*

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

<sup>1</sup> This practice 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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<sup>2</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>3</sup>

- E1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory
- E2917 Practice for Forensic Science Practitioner Training, Continuing Education, and Professional Development Programs

## 3. Terminology

### 3.1 Definitions:

3.1.1 *stub, n*—sample device with an adhesive surface used to collect materials for SEM/EDS analysis.

3.1.2 *characteristic particles, n*—particles that have compositions rarely found in particles from any source other than GSR.

3.1.3 *consistent particles, n*—particles that have compositions that are found in GSR and also arise from other non-firearm sources.

3.1.3.1 *Discussion*—Particles within this group are produced through the operation of a variety of processes, equipment, or devices and can be found in the environment with varying levels of frequency.

3.1.4 *commonly associated particles, n*—particles have compositions that are also commonly found in environmental particles from numerous sources.

3.1.4.1 *Discussion*—When present in addition to particles that are characteristic of, or consistent with, GSR, or both, these particles can be of use in the interpretation of a population of particles and, consequently, the likelihood that that population is GSR. In isolation, however, such particles have little use in examinations for GSR.

3.1.5 *morphology, n; morphological, adj*—referring to size, shape, structure, and texture.

## 4. Summary of Practice

4.1 Particles composed of high mean atomic number elements are detected by their SEM backscattered electron signals

<sup>3</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

and an EDS spectrum is obtained from each. The EDS spectrum is evaluated for constituent elements that could identify the particle as being consistent with or characteristic of GSR (2-4). See Section 9 for discussion on classification of particles.

## 5. Significance and Use

5.1 This document will be of use to forensic laboratory personnel who are involved in the analysis of GSR samples by SEM/EDS (5).

5.2 SEM/EDS analysis of GSR is a non-destructive method that provides (6, 7) both morphological information and the constituent elements detected in individual particles.

5.3 Particle analysis contrasts with bulk sample methods, such as atomic absorption spectrophotometry (AAS) (8), neutron activation analysis (NAA) (9), 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 size, shape, and individual particle identification.

## 6. Sample Preparation

6.1 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.

6.2 The sample collection stub shall be labeled 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.

6.3 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.

6.4 Observe the appropriate procedures for handling and documentation of all submitted samples, for example Practice E1492.

## 7. Sample Area

7.1 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<sup>2</sup> and 506.7 mm<sup>2</sup> respectively.

7.2 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 is not always possible in some systems.

7.2.1 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 have been detected. The decision as to how many particles satisfy the requirements of a particular case should be set out in the laboratory's standard operating procedures.

## 8. Instrument Requirements and Operation

### 8.1 General:

8.1.1 Most commercial-grade SEM/EDS systems should be adequate for GSR analysis given that the criteria set forth in 8.2 and 8.3 are met.

8.1.2 Automated data collection of GSR involves some portion of the data collection being controlled by instrument automation software. The extent to which the SEM and EDS systems communicate and are integrated varies according to the manufacturers and the capabilities of the hardware/software. The system shall have the ability to recall stage locations of particles for verification and software for particle recognition.

### 8.2 Scanning Electron Microscope (SEM):

8.2.1 The SEM, operating in the backscattered electron imaging mode, shall be configured to detect particles down to at least 1.0 µm in diameter.

8.2.2 The SEM shall be capable of an accelerating voltage of at least 20 kV.

8.2.3 SEM systems shall include: a motorized stage with automated stage control.

### 8.3 Energy Dispersive X-Ray Spectrometry (EDS):

8.3.1 The detector shall be configured to produce a resolution of better (less) than 150 eV during analysis, measured or extrapolated as the full width at half the maximum height of the Mn K<sub>a</sub> peak (1).

8.3.2 At a minimum, the EDS spectrum shall be acquired at 20 eV per channel.

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

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

8.3.5 The instrument shall be capable of recording spectra obtained from the analysis of candidate particles. At a minimum, an automated system shall be capable of storing all of the particle location coordinates.

### 8.4 Sample Placement:

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

8.4.2 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 could 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).

### 8.5 Detection and Calibration:

8.5.1 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 **(10)**.

8.5.2 The brightness and contrast settings (low and high thresholds) of the backscattered electron detector system determine the limits of detection and discrimination of particles based on their mean atomic number. Threshold settings for the backscattered electron signal should be done with a suitable reference sample of known origin (often supplied by the EDS manufacturer) or elemental standards at the same instrumental settings used for sample analyses. This reference sample should, if possible, be in the microscope chamber at the same time as the samples to be analyzed.

8.5.3 The backscattered electron detector's brightness and contrast should be set to include the 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 threshold limits for ease of detection. If the beam current is changed or drifts, the brightness and contrast threshold limits, which were based on the previous beam current, could no longer be compatible with the new conditions and should be readjusted. Beam current measurements can be taken with (but are not limited to) 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.

### 8.6 *Quality Control:*

8.6.1 When conducting automated analysis of GSR, special measures have to be chosen in order to meet common quality management demands. The use of control charts or tracking of instrument parameters and quality control metrics, or both, is recommended. Therefore, as minimum conditions:

8.6.1.1 Establish a protocol to confirm appropriate instrument operation parameters prior to sample analysis.

8.6.1.2 Monitor the EDS X-ray energy calibration and SEM beam current stability at least once per batch of samples using appropriate reference materials of known elemental composition such as copper, cobalt, etc.

8.6.1.3 Analyze a reference material with particles of known size range and composition to test the accuracy of particle detection and identification. It is recommended that the reference material has been prepared and mounted in a manner comparable to the collection method in use by the submitting agency. The reference material can be a sample of GSR from a known source. Additional environmental particles may be added to ensure the inclusion or exclusion of particular classes of particles. Alternatively, a synthetic, GSR standard containing particles comprised of the primary elements of interest may be used for this purpose. The frequency of analysis of this sample shall be subject to guidelines set out in the laboratory's standard operating procedures.

8.6.1.4 The incorporation of environmental or control samples into the analytical protocol is recommended in order to monitor the cleanliness of the sample collection or analytical system, or both. An environmental sample can be prepared in

a number of ways: for example, it may be an unused stub that has been prepared contemporaneously with the questioned samples or a sample taken from the sample collection or analytical environments (exposed to the air or as a direct sample collection from clean workspace), or both.

8.6.1.5 Care should be taken when transferring samples to and from the instrument to prevent cross contamination. Tools should be cleaned using a solvent and lint free tissue between samples from different sources.

## 9. Data Analysis

### 9.1 *Definition and Classification:*

#### 9.1.1 *Morphology:*

9.1.1.1 Particles identified as characteristic of or consistent with GSR using this method are often spheroid particles, typically between 0.5  $\mu\text{m}$  and 5.0  $\mu\text{m}$  in diameter; the remainder are irregular in shape or vary from 0.5  $\mu\text{m}$  to 100+  $\mu\text{m}$  in size, or both **(6, 11, 12)**. In general, it is not consistent with the mechanisms of GSR formation to find particles by SEM-displaying crystalline morphology. However, such particles have occasionally been observed in known primer GSR residues. Since morphology can vary greatly, it should never be considered as the only criterion for identification of GSR.

#### 9.1.2 *Elemental Composition:*

9.1.2.1 The elemental composition is the most diagnostic property to determine if a particle could be GSR **(13)**. In some instances, it could provide additional forensic value to compare the elemental composition of the recovered particulate with case-specific known source items, such as the recovered firearm, cartridge cases, or victim-related items.

9.1.2.2 Particles with unusual elemental compositions could be encountered in case work. In this circumstance, the elemental compositions of these particles should be compared to case-specific sources, such as cartridges or ammunition/firearm test fire deposits.

9.1.3 Particles classified as characteristic of GSR will have the following elemental composition:

9.1.3.1 Lead, antimony, barium **(7)**.

9.1.4 Particles classified as consistent with GSR will have one of the following elemental compositions:

9.1.4.1 Lead, barium, calcium, silicon.

9.1.4.2 Barium, calcium, silicon.

9.1.4.3 Antimony, barium **(14)**.

9.1.4.4 Lead, antimony.

9.1.4.5 Barium, aluminum.

9.1.4.6 Lead, barium.

9.1.5 Particles classified as commonly associated with GSR will have one of the following elemental compositions:

9.1.5.1 Lead.

9.1.5.2 Antimony.

9.1.5.3 Barium.

9.1.6 The following compositions have been observed from different kinds of ammunition with "lead-free/non-toxic" primers **(15, 16)**.

9.1.6.1 Particles that have a composition characteristic of GSR, will have one of the following elemental compositions:

(1) Gadolinium, titanium, zinc **(17)**

(2) Gallium, copper, tin **(17)**.