



**Guide for Primer Gunshot Residue Analysis by
Scanning Electron Microscopy/Energy Dispersive
X-Ray Spectrometry 11-29-11**



GUIDE FOR PRIMER GUNSHOT RESIDUE ANALYSIS BY
SCANNING ELECTRON MICROSCOPY / ENERGY DISPERSIVE X-RAY SPECTROMETRY

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PREFACE

The Scientific Working Group for Gunshot Residue (SWGGSR) was established in 2007 and funded by the National Institute for Justice (NIJ). Currently, the group comprises 27 experienced gunshot residue analysts from the United States, as well as from Australia, Canada, Finland, Germany, Israel, and the United Kingdom.

The purpose of SWGGSR is to make recommendations for internationally accepted guidelines for the forensic examination of gunshot residue. This document is the product of input from subcommittees within the SWGGSR group. Its intended use is as a comprehensive guide to the forensic laboratory practitioner, the legal community, and academia. While no guide can address all issues in every specific situation, the guide represents the mutually agreed upon opinions of the expert group.

This guide covers the analysis of primer gunshot residue (GSR) by scanning electron microscopy/energy-dispersive x-ray spectrometry (SEM/EDS) by automated and manual methods. It makes recommendations concerning interpretation and report writing, contamination issues, expert witness testimony, and training. It provides detailed background information concerning ammunition and its contribution to gunshot residue particles. The guide also contains a glossary of SEM terms and a list of published reference materials.



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INTRODUCTION

Gunshot residue (GSR) particles form as a result of rapid cooling of the discharge gases and solid matter, originating from partially reacted components of the primer and propellant, as well as from the metallic components of the ammunition and firearm. In addition, GSR originating from ammunition previously discharged from the firearm can also contribute to GSR ejected during a subsequent discharge.^{1 2 3}

Some of the gases condense in the form of spheres, but they also interact with solid residue materials to form complex mixtures and aggregate forms. Although some residue material can be ejected with little or no physical or chemical modification, most residue particles show evidence of exposure to or formation at extremely high temperatures and pressure.

Spheroidal particles range in size from sub-micrometers (μm) to several hundred micrometers in diameter. Irregular and aggregate particles generally constitute the majority of larger GSR particles produced, ranging in size typically from a few micrometers to several hundred micrometers.

This guide pertains to the detection of elemental residues originating from

- Residues formed through the explosive reaction of the primer compounds
- Material originating from the bullet and bullet jacket or coating
- Material eroded from the cartridge case, primer cup and other cartridge components
- Materials originating from the interior of the firearm chamber and barrel, including residues originating from previous discharges of the firearm and foreign materials, such as metal oxidation/corrosion, soil, and debris can exist

The elemental primer components are the primary source of particles used for the identification of GSR.

There are several classes of primers and within each class there is variation in the composition.

Secretions and other debris from the subject's skin or hair can also contribute to the physical form and chemical composition of GSR particles collected.

The combination of Scanning Electron Microscopy and Energy Dispersive Spectrometry (SEM/EDS) has widespread scientific acceptance as the optimum technique for the examination and analysis of GSR^{4 5} for the following reasons:

- The technique is non-destructive.
- Minimal sample preparation is necessary.
- Individual particles can be analyzed.



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- The morphology of particles can be examined.
- GSR can be identified with a high level of confidence on the basis of the elemental composition and morphology.
- Highly effective and rapid automated systems have been developed for particle detection and classification.

SEM/EDS can be applied either manually or in an automated mode to the search for and analysis of potential GSR particles. In both manual and automated search modes, the sample surface is scanned in a systematic, defined sequence. Automated search is the preferred method due to increased efficiency.

In a manual search, the search regime and instrument parameters are determined by the operator.

In an automated search, the user sets a number of parameters that determine the minimum particle size to be detected as well as the area of the sample surface to be searched.^{6 7 8}

However, once these parameters have been set, the search mode is fully controlled by the search software using a combination of controlled movements of the automated stage and electron beam positioning.

In both manual and automated systems, the search targets particles of high mean atomic number. These are visualized using a Back Scattered Electron (BSE) detector set at a threshold for high atomic number or calibrated to provide a correlation of atomic number with gray scale. Once a particle with a bright BSE image is detected, it is analyzed by EDS.^{9 10} In the automated search mode, this process is controlled by the search software. The automated search software classifies the composition of each particle analyzed and saves the coordinates or position on the sample stub for later review. In many systems, a low-resolution image of the analyzed particle is saved for later inspection.

Multiple sample analysis is possible with current, automated GSR search systems.

This guide applies to SEM/EDS analysis of GSR samples taken from human subjects,¹¹ clothing,^{12 13} and objects allegedly associated with shooting incidents. Adhesive sample mounts on round, 12.7 mm diameter, aluminum stubs are most commonly used for the examination of samples taken for the purposes of detection of GSR, although smaller and larger samples can be accommodated. In some jurisdictions, rectangular sample tabs are used.

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.



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Propellant residues are not directly detected using this method. However, as partially burnt propellant grains can be associated with primer and bullet residues, propellant particles can be detected indirectly. In order to identify propellant detected in this manner, the analyst must recover the particle from the sample stub and analyze it using suitable techniques such as Fourier Transform Infrared Spectroscopy, Raman Spectroscopy, and/or Chromatographic techniques.¹⁴

INSTRUMENTAL REQUIREMENTS

In analytical SEM, the electron beam must be coherent and of high current density for efficient production of x-rays. There are three main systems for generating electrons in SEM used in analytical systems:

- 1) Tungsten filament/electron gun
- 2) Lanthanum hexaboride (LaB_6) filament/electron gun
- 3) Field Emission Gun

All are suitable for both manual and automated GSR analysis. Both LaB_6 and Schottky Field Emission Guns provide extended filament life and stability with superior image resolution to that of a tungsten filament electron gun.

Accelerating voltages of at least 20kV are required for optimum generation of BSE and x-rays from GSR particles. Commonly, an accelerating voltage of between 20 and 25kV is used for manual and automated searching.

The BSE detector must be highly sensitive to changes in atomic number. For manual searching, the BSE detector should be capable of operating at rapid scan rates resulting in little or no image streaking with movement of the sample stage. Large area, passive, scintillator detectors have excellent atomic number contrast and operate exceptionally at fast scan rates. However, solid state BSE detectors now provide superior atomic number contrast with the scan rate response approaching that of scintillator detectors. Consequently, either type is suitable for GSR analysis.

Using suitable search parameters, the SEM should be capable of detecting particles approximately 0.5 μm in diameter.

In SEM utilizing manual or automated searching for GSR, the ability of the instrument to detect sub-micrometer sized particles is determined by adjustment of the beam current, spot size, working distance, BSE detector sensitivity (brightness/contrast threshold setting), image magnification, and the stage movement. A known standard that includes suitable sub-micrometer sized particles is employed to ensure the SEM/EDS system is functioning properly.



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The SEM beam stability is crucial for a stable BSE signal and is critical in both manual and automated systems.

The EDS energy calibration is important for accurate identification of elements and, hence, automated classification of particles.¹⁵ The SEM beam stability and EDS energy calibration can be monitored during analysis by some automated systems, using a suitable reference standard. A performance standard can be run to check and monitor the operating conditions of the SEM. (see Calibration & Quality Assurance)

In automated GSR particle search systems, the extent to which the SEM and EDS communicate and are integrated varies according to the manufacturers involved and the capabilities of the hardware/software architecture.

Automated GSR/particle search systems may include

- A motorized and programmable sample stage
- Automated stage and beam control with the ability to recall stage locations for verification of the composition of particles of interest
- A saved image of the analyzed particle
- Particle detection and classification software (this software can be housed in either the SEM or the EDS)

Most EDS instruments will be suitable for the analysis of GSR particles detected during manual searches. There are a number of EDS instruments that incorporate or can be interfaced with automated GSR particle search systems.

EDS systems capable of analyzing GSR particles, typically, will

- Be capable of resolving clearly the Sb $L\alpha_1$, $L\beta_1$, and $L\beta_2$ and the Ba $L\alpha_1$, $L\beta_1$, and $L\beta_2$ peaks. Instruments having an x-ray line resolution of less than 150 eV, measured as the full width at half the maximum (FWHM) height of the Mn $K\alpha$ peak, will be capable of resolving these lines; however, instruments having a resolution of approximately 130 eV for the Mn $K\alpha$ peak will more clearly resolve close peaks, such as the Ca $K\alpha$ and Sb $L\alpha_1$ peaks.
- Have a calibrated, scaled display of x-ray energy versus counts
- Have the ability to identify and label x-ray lines and a facility for hard copy output of the display contents.

A display resolution of 1024 channels at 20 eV per channel is suitable for visually interpreting the x-ray spectrum and identifying the elements present; however, a setting of 2048 channels at



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10 eV per channel displays the x-ray lines at greater resolution and, hence, provides clearer differentiation of very close x-ray lines such as Sb $L\alpha_1$ and Ca $K\alpha$ and Ba $L\alpha_1$ and Ti $K\alpha$.

The spectral range of the EDS display and output must encompass the x-ray lines of relevance to the identification of GSR. A range of 0-20keV is recommended although a minimum range of 0-15 keV will suffice.

Windowless EDS detectors are not suitable for GSR analysis as samples are often contaminated with biological and other organic matter that can out-gas and deposit on the unprotected detector surface resulting in a decline in performance. Beryllium or organic/polymeric windows are suitable for protection of the detector.

Automated systems will also include software capable of acquiring x-ray spectra for a specified collection time or number of x-ray counts. It is desirable that the spectrum obtained from the analysis of each particle detected be stored. At a minimum, an automated system must be capable of storing all of the particle co-ordinates so that the particles can be relocated for confirmation.

CALIBRATION & QUALITY ASSURANCE

A protocol must be established to confirm optimal operating parameters on a routine basis. This can be facilitated by the use of appropriate standards or reference samples.

The accuracy of the SEM's dimensional measurement function at various magnifications can be calibrated using a suitable traceable standard periodically and a record kept in a permanent log. Note that for GSR analyses, the accuracy of the scale marker is not a critical measurement, and, hence, calibration of the SEM measuring scale is not mandatory.

To ensure that the SEM is operating at optimum performance, there are a number of procedures that should be followed:

- *Saturation of the filament* is optimized to the accelerating voltage used for the analysis. A balance needs to be obtained in setting the filament current. Too low of a current will make it insensitive to small changes, but too high of a current will reduce the filament life. Some instruments include automatic saturation, but manual saturation can provide better performance.
- *Centering the filament and/or setting the gun shift/tilt controls* optimizes image quality and beam current.
- *Centering the aperture* using focus wobble or lens modification. Centering this aperture allows the electron beam to pass through the center of the objective lens for best resolution and image quality.



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- *Astigmatism/focus correction.* With no astigmatism, the sharpest image is obtained at a focused point.
- *Degaussing/anti-hysteresis* prior to setting SEM parameters. Elimination of hysteresis of the magnetic field optimizes accuracy of the magnification and alignment of the electron optical axis.

The EDS detector should be calibrated regularly. The frequency of the calibration is determined by the energy drift of the detector. A record of the calibration check should be kept according to laboratory protocol. Automatic methods for calibration are described in documentation from the manufacturer.

The spectral resolution of the EDS should be determined regularly, depending on the stability of the instrument. This can be determined automatically or manually by measuring the width of the Mn K α , Co K α , or Cu K α peak at half the maximum peak height .

An automated system utilizes a known atomic number standard or standards to adjust the BSE detector. Similar procedures should be used to set the BSE parameters for manual searching of GSR. The standards used to set the operating parameters for an automated search are generally stipulated by the automated search system that is employed. These standards can simply be used to set a minimum atomic number detection threshold for the BSE detector, or they can be used to adjust the BSE detector gray levels to particular values of atomic number.

When operating in automated mode, a suitable reference sample/standard with a known particle distribution and/or composition, size and location should be analyzed in order to test the accuracy of particle detection and classification. This standard should be analyzed regularly to check the detection efficiency of the instrument. It can also be suitable for adjusting the BSE detector and setting the SEM/EDS parameters prior to automated or manual analyses.^{16 17}

Suitable standards include

- ENFSI (or Plano) Synthetic GSR Standard
- A sample prepared from known GSR and subjected to repeated analysis to establish the distribution and compositions of the particles

A stub that has not been used for collection of evidence samples (a negative or environmental control) should also be included with each sample set to be analyzed. Environmental controls can be used to test the integrity of the operating procedures by monitoring environmental particles and potential contamination within the sampling and/or analytical system. An environmental control is a sample stub exposed to any process in which GSR sample stubs experiences.

Examples include sample preparation areas, carbon-coating equipment, or inside the SEM chamber. When a laboratory or a commercial company manufactures sampling stubs/kits, it is



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recommended that at least one unused/blank sample stub from each batch be analyzed in order to establish its acceptability and performance. It is suggested that ISO 9000 companies provide the sampling kits.

PROCEDURE

Sample Preparation

The work area and tools used for the preparation and examination of samples should be free of all materials that could transfer to the sample surface. Exposure of the sample collection stub should be limited to essential procedures only and it should be replaced into its container immediately upon completion of the task.¹⁸

The collection stub should be labeled in such a manner that it can be distinguished from the other samples being examined. In doing so, extreme care should be taken to avoid contamination of the sample surface.

The samples can first be examined with a stereomicroscope to examine the material adhering to the sample. This preliminary examination can, for example, detect the presence of partially burnt propellant grains or other particles of interest.

The surface of the sample must be coated with a conductive material if a non-conductive adhesive is used for the sample collection unless an environmental SEM or variable pressure/low vacuum instrument is used for the analysis.

Carbon is the recommended coating material, as it provides a negligible backscattered signal and does not emit interfering x-ray lines. The coating should be uniform and of adequate thickness to eliminate charging of the sample during analysis. If charging occurs, the specimen should be re-coated and re-analyzed.

It should be noted that some materials can charge even when conductive adhesive has been utilized (e.g. samples loaded with fibers). In such cases, the sample should be coated to achieve an effective analysis.

Setting the Detection Parameters

The working distance, electron beam spot size and magnification should be set at the optimum settings for detection of GSR by a particular instrument. These settings should be fixed for all analyses unless there is a particular reason for modifying the parameters. Such changes should be documented in the relevant case record(s).

GSR particles originating from the most common ammunition types are detected as a result of their bright BSE image. The intensity of this image is related to the electron beam current, mean



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atomic number, and size of the particle. The magnitude of the brightness of the BSE signal from a particle increases in intensity as the beam current increases.¹⁹

The beam current can 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.

The sensitivity of the BSE detector to changes in mean atomic number of a particle at a given working distance, spot size, and beam current is adjusted using the brightness and contrast settings. Using an appropriate reference or calibration sample, the BSE detector response should be adjusted to set a threshold or gray scale that will allow the detection of a particle whose mean atomic number is like that of a typical Pb, Ba, Sb particle. These same instrument parameters should be used during the analysis of the questioned samples.

In most current automated systems, the BSE detector is set to a gray scale with a relatively broad atomic number detection range using a multiple element sample. This usually incorporates a procedure for measuring the beam current.

It should be noted that some brands of environmentally safe ammunition contain no heavy metals in the primer. Particles generated from such ammunition types will not contain high atomic number elements. As such, these particles will not be readily detected using typical operating parameters of the SEM for detection of traditional heavy-metal GSR.

Automated Analysis

Automated analysis of GSR involves some portion of the analysis being controlled by preset 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. Software control of the instrument enables a systematic and comprehensive search of a predetermined area of the sample.

When an automated SEM/EDS system is employed, data collection from the entire surface area of the sample collection stub is recommended, subject to limitations of the search software and/or sample geometry. Where such limitations exist, it is recommended that as much surface area as possible be analyzed.

Depending on the requirements of the examination, a threshold on the number of detected particles that have been classified as GSR (or other composition of interest) can be applied, resulting in the termination of the particle search before the end of the programmed search area.

An automated SEM/EDS system should provide hard copy output and long term storage of the sample identifier and stage position, the field of analysis, the stage coordinates of all detected



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particles, the total number of particles detected, and the total number of particles classified as GSR.

Particles classified as potential GSR by the automated search software must be relocated and the x-ray spectrum reacquired. Where large numbers of particles of potential GSR are detected, the re-analysis and verification of only a reasonable number of the total particles detected can be sufficient.

Manual Examinations

Manual examination of the total surface area of the sample can be prohibitively time-consuming. However, since particles are collected in a random manner on the sample surface, it is reasonable to analyze a portion of the stub surface by employing an appropriate analytical protocol to establish a suitable sub-area to search. Selection of a small search area can significantly decrease the probability of detecting particles of interest.²⁰ Particles should be analyzed if their backscattered electron signal brightness exceeds the desired threshold setting.

Analysis of Detected Particles

The potential GSR particles detected by automated analysis are relocated and confirmed by acquiring an x-ray spectrum from each particle using a small area analysis positioned completely within the particle's volume (generally centered on the particle). The spectral acquisition must provide an adequate statistical basis for identification of all peaks of interest²¹ by accumulating sufficient x-ray counts at the desired count rate (a dead time of between 20% and 50% is optimum). However, if a brief spectral acquisition indicates that the composition is not characteristic of, or consistent with GSR, acquisition may be stopped.

When no GSR-related particles are detected on a sample, the analyst should ensure that the focus and general operating conditions for the sample have not varied and are correct. At high accelerating voltages, the interaction of the beam within a particle can be large. This needs to be considered when analyzing small particles as the interaction volume can exceed the size of the particle.

Where the BSE image indicates that the particle is non-homogenous, the variation in composition within the particle should be characterized by multiple spot or small area analyses of the various phases. Alternatively, x-ray mapping may be used to demonstrate the distribution of the component elements.²²

Samples from Ammunition & Firearms

Although the identification of the brand of ammunition used from the composition of the detected GSR particles is rare, it can be relevant to the investigation to establish whether the



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particles detected were consistent with having originated from the ammunition used in the crime.^{23 24} More importantly, it can be possible to exclude detected particles as having originated from the ammunition and firearm that was used. In doing so, the analyst must be aware that GSR originating from previous shootings from a firearm can contribute to GSR from later shootings from that firearm. If this is an issue in the case, a sample can be collected from the interior of the fired cartridge case. There are a number of ways this can be achieved including but not limited to the following:

- Washing the interior of the cartridge case using a few drops of volatile liquid, such as methanol, ethanol or isopropanol and transferring the resultant suspension of GSR particles to a sample stub drop wise using a disposable pipette or syringe. The solvent is evaporated to dryness between applications.
- Using a toothpick or wooden applicator stick to transfer particles from the fired case or firearm barrel directly to a prepared collection stub.
- Covering the opening with the adhesive side of a prepared stub, inverting the two, and giving a firm tap to the base of the cartridge case.
- Lightly wiping a dry cotton swab inside the cartridge case, then rolling the cotton bud onto the surface of an adhesive sample stub. Note that this technique will result in many cotton fibers also being transferred onto the stub.

If the firearm believed or known to have been used in the shooting is available, a second sample can be taken from the breech area or muzzle, if it has not already been test fired. This can be done using a toothpick, a wooden applicator stick, or a dry or wet (water, methanol, ethanol or isopropanol, for example) cotton swab stick as described above. Consideration may also be given to test firing the known ammunition from the firearm after all other sampling has been done. Sampling directly from the firearm can also establish the contribution of residues originating from previous firings of different ammunition.²⁵

If the firearm used in the crime is not available, it may be possible to obtain samples pertaining to the shooting from

- The gunshot wound area
- The bullet itself²⁶
- An impact site
- The surface believed to have been adjacent to the point of discharge

If the above options are not possible, it could be necessary to obtain GSR samples by discharging the same type of ammunition and firearm.



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Care should be taken to examine a wide range of particles obtained from the cartridge case and firearm or discharge test, including particles that fall within the same range of size and morphology as composition can vary significantly with size and morphology.²⁷

DOCUMENTATION

The following documentation is required for significant particles reported in the examination by either automated or manual analyses:

- SEM images of relevant particles showing their morphologies. Images of particles rejected as being GSR may also be included, particularly those that have similar elemental profiles to GSR but were rejected due to additional elements rarely found in GSR and/or inconsistent morphology. A scale bar or particle measurement should also be included with the SEM images of relevant particles.
- X-ray spectra of the relevant particles. All elements present should be clearly identified.
- The labels on both images and spectra must clearly identify their association and source.
- The operator should follow other intra-laboratory protocols for documentation as appropriate.

REPORTING CRITERIA

To better understand GSR it is necessary to have a knowledge of the sources of these residues: primarily, the ammunition and the firearm. (See Appendix B Ammunition)

Classification of GSR

In the following classification scheme, individual particles are classified as either *characteristic* of, *consistent* with or *commonly associated* with GSR. The most diagnostic property used to determine if a particle is characteristic of or consistent with GSR is its elemental profile.²⁸

Particles classified as *characteristic* of GSR have compositions rarely found in particles from any other source (see “*Sources of Particles with Compositions Similar to GSR*”).

Particles classified as *consistent* with GSR have compositions that are also found in particles from a number of relatively common, non-firearm sources. Particles within this group are



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produced through the operation of a variety of processes, equipment or devices and can be found in the environment with varying levels of frequency.

Particles classified as *commonly associated* with GSR have compositions that are also commonly found in environmental particles from numerous sources. However, when present, *in addition to* particles that are characteristic of, and/or consistent with GSR, these particles can be of significance in the interpretation of a population of particles and, consequently, the likelihood that that population is GSR. In isolation, however, such particles have little significance in examinations for GSR.

Particles that are characteristic of GSR:

Ammunition with Sinoxid-type Primers

Almost all GSR that is classified as *characteristic* in elemental composition is derived from ammunition incorporating primers based on the “Sinoxid” formulation that contain lead styphnate (possibly with other lead compounds), antimony sulfide and barium nitrate.²⁹ Detonation of these primers generates numerous particles considered to be characteristic of having originated from this type of primer. These particles must contain

lead, antimony, and barium

Numerous additional components can be added to this basic primer composition; each of which can contribute to the elemental composition of GSR particles originating from the particular ammunition. In addition, other elements originating from the other ammunition components and firearm can contribute to the elemental profile of GSR particles.

In determining the relative levels of components in a mixture, Goldstein et al stated: “The terms ‘major,’ ‘minor,’ and ‘trace’ as applied to the constituents of a sample in this discussion are not strictly defined and are therefore somewhat subjective.” Because quantitation using EDS cannot be applied to particles having complex morphology, the height of the major X-ray line for a particular element can be used as an approximate measure of its concentration. Consequently, the criteria postulated by Goldstein et al may be employed using peak height relative to the major X-ray line/peak of the most prevalent element as a determinant of the relative abundance of each component³⁰. Note for the purpose of GSR analysis, carbon should not be used as the most prevalent element in peak height comparisons when using a light element detector. Major elements are considered those whose most intense x-ray peak is greater than approximately 30% of the highest peak (relative peak height). Minor elements are those whose most intense x-ray peak is less than approximately 30% but higher than approximately 10% of the highest peak



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(relative peak height). Trace elements are those whose most intense x-ray peak is less than approximately 10% of the highest peak (relative peak height).³¹ For example, if a barium $L\alpha$ peak is the tallest major peak in a spectrum, a sulfur $K\alpha$ peak of approximately the same height would be major; an intensity that is approximately the height of the barium $L\beta$ peak would be minor; and an intensity that is approximately the height of the barium $L\gamma$ peak would be trace. It should be noted that the determination of low levels of sulfur in the presence of large amounts of lead is difficult due to the closeness of the sulfur K lines and the lead M lines resulting in poor resolution of these lines by EDS.

The following elements can be found in the above characteristic particles as major, minor or trace components: (refer to Glossary)

silicon, calcium, aluminum, copper, tin

The following elements can also be found in the above characteristic particles, usually as minor or trace components:

iron, sulfur, zinc, potassium, chlorine, phosphorus, nickel

In addition, there are some elements that are rarely found in GSR derived from “Sinoxid” type primers but, when present, can provide a strong link to a specific ammunition or sub-type of ammunition. These include the following elements:

cobalt, zirconium

One or more of the above elements may be present in these characteristic GSR particles depending on the ammunition and firearm that they originated from. It should be noted that this is not a comprehensive list and other elements have been identified in GSR.

Ammunition having calcium silicide based primers incorporating a Tin Foil in the Primer

Older Sellier, Bellot, and Prague (SBP headstamp) produce an antimony-free primer containing lead styphnate, barium nitrate and calcium silicide. This ammunition also incorporates a tin foil disc that seals the primer into the primer cup. Discharge of this brand of ammunition produces the following particles that are characteristic of GSR originating from these ammunitions:

*lead, barium, calcium, silicon, and tin*³²

Other components may also be present as the primer composition shows variation between calibers and the date of manufacture. Consequently, GSR particles originating from S&B ammunition can also contain one or more of the following other elements:



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copper (as either a major, minor or trace component), and *iron, sulfur, zinc, potassium and chlorine*, usually as minor or trace components.

Tagged Ammunition

Some ammunitions manufactured specifically for use by police in some European countries are tagged with specific elements.^{33 34} These incorporate “Sintox,” environmentally safe primers as well as taggant elements. In “Sintox” primers 2,4-dinitrophenol (also known as Dinol or Diazole) and other heavy-metal-free compounds replace the compounds of lead, antimony and barium. The presence of taggant elements added to the ammunition gives rise to the formation of characteristic particles when discharged. Two sources of tagged ammunition are used.

★ **Ammunition Manufactured by RUAG Ammotec AG**

Particles that have a composition characteristic of GSR generated by RUAG Ammotec AG ammunition will have the following elemental profile:

Gadolinium, titanium, zinc

Particles originating from this ammunition may also contain trace levels of calcium and sulfur.

★ **Ammunition Manufactured by MEN GmbH**

Particles that have a composition characteristic of GSR generated by MEN GmbH ammunition will have the following elemental profile:

Gallium, copper, tin

Particles originating from this ammunition may also contain trace levels of potassium and sulfur.

Particles that are Consistent with GSR:

Not all particles formed as a result of the detonation of “Sinoxid”-type primers have characteristic lead-antimony-barium composition. Particles containing one or a combination of two of the three elements (lead, antimony and barium) can also form.

In addition, many non-Sinoxid primers based on lead styphnate and other lead compounds do not contain both antimony sulfide and barium nitrate. Furthermore, some “lead-free” ammunition contains barium nitrate and antimony sulfide. As such, ammunition containing these types of primer will not generate characteristic lead-antimony-barium particles.

Consequently, particles with the following elemental profiles are considered to be consistent with GSR:



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- *Barium, calcium, silicon* (with no more than a trace of sulfur)
- *Antimony, barium* (usually with no more than a trace of iron or sulfur)³⁵
- *Lead* with levels of antimony greater than trace amounts
- *Barium, aluminum*
- *Lead, barium*
- *Lead, barium, calcium, silicon* (produced by antimony-free, lead styphnate, barium nitrate, and calcium silicide based primers like Hirtenberger)

There are many (non-tagged) variants of primers incorporating 2,4-dinitrophenol that are classified as “Sintox” primers or “environmentally safe.”

Particles with the following elemental profiles are considered to have a composition consistent with GSR formed from all ammunitions incorporating “Sintox” primers:

- *Titanium, zinc*^{36 37 38}

Particles with this composition may also contain copper or tin (e.g., from jacketing material), and/or silicon, calcium, and aluminum.

Particles having the following elemental profiles are considered to have a composition consistent with GSR formed from some brands of “environmentally safe” ammunition:

- *Strontium*^{39 40 41}

Particles with this composition may also contain copper (e.g., from jacketing material), aluminum, potassium, calcium, and/or barium.

Particles that are Commonly Associated with GSR:

Commonly associated particles with gunshot residues could include

- Lead with only trace levels of antimony
- Lead
- Antimony
- Barium (in the absence of sulfur)

These particles may also contain one or more of only the following other elements: silicon, calcium, aluminum, copper, and trace amounts of iron, sulfur, phosphorus, zinc, nickel (in conjunction with copper and zinc), potassium, chlorine, and tin.



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Additional Particle Compositions

Additional particle compositions can be classified according to the aforementioned systems. Other kinds of primer compositions could include mercury fulminate based primers for example.⁴² This can aid in differentiating GSR from environmentally or occupationally produced particles. However, any additional classification of particle compositions must be subject to validation and peer review.

Sources of Particles with Compositions Similar to GSR:

There are a small number of (non-firearm) sources of particles having compositions similar to GSR derived from “Sinoxid” type primers (that is, particles containing lead, antimony, and barium). Each of these sources produces a wide variety of particles that are characteristic of the particular source. Almost all of these particles can be clearly distinguished from residues originating from the discharge of a firearm by their morphology and/or composition. However, an extremely small number of particles originating from such sources can be very similar to or indistinguishable from GSR. It is extremely unlikely that, out of all the particles generated by any of these devices, only particles having a similar composition to GSR would be detected.

Cartridge/Primer-Operated Tools & Devices

There are a number of tools and devices that incorporate a primer to initiate a reaction or action critical to the purpose of the device. These include starter pistols (blank cartridges),⁴³ signal guns and flares,⁴⁴ cartridge-operated nail guns,^{45 46} hand grenades,⁴⁷ tasers,⁴⁸ and some types of air bags deployed in vehicles.⁴⁹

Many of these have primers that do not produce particle populations with compositions that can be classified as characteristic of GSR. Furthermore, residues originating from these sources are characterized by the presence of large numbers of particles derived from other components of the device.

However, there are several specific cases in which a particle with a composition that is characteristic of GSR can be produced:

Cartridge-Operated Nail Guns and Fasteners

Cartridge-operated nail guns and fasteners use the explosive energy of a blank cartridge (powder load) discharge to propel a nail into the work piece. Most nail guns use rimfire cartridges that are based on lead-barium component primers (antimony-free), although tools available in the 1980s



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under the “Obo” brand, incorporating “Gevelot” brand cartridges,⁵⁰ did employ a primer based on the “Sinoxid” formulation. This product is no longer available worldwide and may not have been imported into many countries. Hence the possibility of encountering residues originating from this tool is extremely low.

Fiocchi (Italy) brand cartridges that were available in 1999, used in conjunction with Bossing brand cartridge-operated tools were also reported to produce characteristic lead-antimony-barium particles.⁵¹

The use of such cartridge operated tools varies considerably from country to country, is subject to local industrial and health and safety regulations, and in some places their use has largely been supplanted by compressed air operated systems.

In North America, revisions to OSHA regulations (OSHA Standard 1926.302(e)(12)) in 1993 mandated adherence in the workplace to the ANSI Standard, A10.3-1970, “Safety Requirements for Explosive-Actuated Fastening Tools” This standard requires that only the minimum powder load needed to drive the fastener be used. In practice this greatly restricted the use of high velocity tools in favour of the use of low velocity piston based tools using rimfire based cartridges of .22, .25 or .27 caliber.

When the use of cartridge-operated tools is indicated, the analyst is advised to collect residue samples from the device for comparison purposes.

Air Bag Devices

Some vehicle air bags incorporate a primer device that is mounted in the passenger side dashboard of a vehicle. They employ primer initiation to guarantee a sufficient rate of inflation. Most of the air bag primer residue particles obtained from these deployed passenger side devices contain elements unusual to GSR as well as lead, antimony, and barium. Furthermore, many thousands of non-primer particles, characteristic of air bag residues but foreign to GSR, are also expelled from the air bag on deployment.⁵²

Fireworks & Other Pyrotechnics

Lead, antimony, and barium compounds are rarely found together in fireworks and pyrotechnics;⁵³ ⁵⁴ however, few uncommon firework products, such as the “Crackering Ball”, can contain all three elements.⁵⁵ The majority of particles generated from the ignition of firework products contain elements not typically found in GSR, such as magnesium and/or other elements present



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in levels not generally seen in GSR (for example, elevated levels of chlorine and potassium). A very small proportion of particles generated, however, have simple three-component (lead, antimony, and barium) compositions. The morphology of firework particles commonly shows the characteristics of having been formed in very high temperatures.^{56 57} The rare instances of firework particles found with lead, antimony, and barium have not been found with spheroid morphology

Pyrotechnic mixtures that contain aluminum powder and barium nitrate (in addition to various other compounds) can give rise to some spherical particles containing barium and aluminum.⁵⁸ Such particles are also commonly generated from firearm ammunition primers rich in aluminum and barium nitrate. A small proportion of the aluminum-barium particles generated by pyrotechnics can be indistinguishable to those generated by certain ammunition.

Brake Pads

Brake pads containing compounds of lead, antimony, and barium have been used by some vehicle manufacturers.^{59 60} The friction caused by the application of the brakes results in the shedding of fragments and particles from the brake pads. Some of these particles contain lead, antimony, and barium, usually with a variety of other elements. Most particles can be clearly distinguished from GSR due to the presence of elements rarely found in GSR, and/or elements present in levels not generally seen in GSR, such as elevated levels of iron.

Most of these particles are irregular in form and heterogenous in composition (that is, separate lead, antimony, and barium zones are generally observed). However, these particles can resemble GSR when the other elements that are characteristic of the brake pads are absent and friction results in more intimate mixing of the lead, antimony, and barium components.

The use of lead in brake pads was discontinued for occupational health and environmental reasons in the early 2000s. Vehicles that still have such brake pads will be less common as time goes by.

Non-GSR Sources of Particles having a Composition Consistent with GSR:

There are a number of relatively common, non-firearm related sources of particles that can have compositions similar to particles classified as *consistent with* GSR: that is, particles containing lead and antimony, lead and barium, or antimony and barium. In most cases, the morphologies of particles from these sources are irregular, angular, or crystalline. As such, the morphology of many of these particles does not support a GSR origin. In addition, most of these particles will contain elements not consistent with GSR or high levels of elements usually found in only trace amounts in GSR.



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Lead-barium-calcium alloys are used as bearing metals. Lead antimonate [$\text{Pb}_3(\text{SbO}_4)_2$], Naples Yellow, is a pigment in artists' paint.

Antimony barium tartrate has been used as a gapeworm treatment for birds.

Some red paints tinted with red lead pigment (lead tetroxide) and lithopone (barium sulfate) co-precipitated with antimony red (antimony pentasulfide) have been encountered. Particles derived from such paint consists of discreet particles of lead tetroxide associated with discreet particles of barium sulfate/antimony pentasulfide bound by paint binder.

Lead-antimony alloys are used in lead annealing of steel wire; sinkers and weights in fishing; weights for scuba diving; capping for wine bottles; flashing and damp coursing; lead-acid battery electrodes; X-ray shielding; vessels and plumbing in the manufacture of corrosive gases and liquids; and antique toys and models. Lead-antimony particles from these sources tend to have only a trace amount of antimony and morphologies not consistent with formation at high temperatures.

Two-component particles having compositions and morphologies consistent with GSR can be found in the above mentioned examples of non-GSR sources of particles with compositions characteristic of GSR. Nevertheless, those sources can be eliminated if the particles contain the presence of elements (such as Mg) rarely found in GSR or the population of particles in the sample reveal elements (such as Fe) present in levels not generally seen in GSR.

Further Studies

The environmental sources of GSR-like particles are subject to further evaluation and studies.

INTERPRETATION

A comprehensive analysis of the entire population of particles detected is necessary to establish the probable origin of any potential GSR particles within that population. These particles should be assessed on the basis of **composition** and **morphology**. The probability that a population of particles is GSR increases significantly with

- The number of particles characteristic of, and consistent with GSR
- The presence and number of particles commonly associated with GSR



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- The absence of elements or levels of elements in the detected particles that are not commonly found in GSR
- The absence of accompanying particles characteristic of a source of non-firearm, GSR-like particles
- The presence of morphologies that indicate formation at highly elevated temperatures.

Note that the morphological features of particles smaller than approximately 1 μ m are very difficult to discern with most instruments.

The presence of multiple *characteristic* particles as well as other particles *consistent* with GSR is generally sufficient to provide unequivocal identification of these particles as GSR.

An approach to the identification of particles considered as characteristic of or consistent with GSR is to compare the elemental profile of the recovered particulate with that collected from case-specific known source items, such as the recovered weapon, cartridge cases, and/or victim-related items whenever necessary. GSR particles with non-routine elemental profiles including the presence of additional elements can be encountered in case work. The overall elemental profile of the GSR particle population should be consistent between the questioned and known source samples.⁶¹

Reporting Considerations

There are a number of considerations that must be taken into account in reporting the results of a GSR examination:

- The likelihood that the detected particle(s) is/are GSR
- The number of detected particles having compositions that are characteristic of and/or consistent with and/or commonly associated with GSR
- The definitions of “characteristic” and “consistent” when used to describe the compositions of the detected particles with respect to GSR
- The extent to which the detected particle(s) can be distinguished from non-firearm sources of particles having GSR-like compositions



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- Whether the particles were consistent or different in composition to GSR originating from the ammunition and/or firearm used in the shooting
- Where very few GSR particles are detected, the following statements can be considered:
 - Very little (or no) interpretation can be applied to finding so few particles.
 - Low levels, especially single particles, have on occasion been found in the environment and on police officers who have recently handled or fired a gun.

The significance of a positive finding is generally consistent with

- The circumstances by which GSR can be deposited onto hands, clothing or other objects include
 - discharging a firearm
 - being in the proximity to the discharge of a firearm
 - coming into contact with a surface that has GSR on it
- The extent to which the examination can or cannot determine the relative likelihood of how or when the particles were deposited onto hands, clothing or other objects.

The significance of a negative finding is generally consistent with

- The subject not having discharged a firearm
- The loss of GSR particles⁶² as a result of washing or physical activity
- The loss of GSR particles as a result of environmental conditions such as rain or wind
- The firearm having been of a type that does not eject significant amounts of gunshot residue onto the hands and/or clothing of the shooter
- The ammunition type producing particles that are not readily detected by SEM/EDS analysis
- A physical barrier preventing deposition of GSR
- The sampling procedure did not result in the collection of GSR particles



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- Some GSR particles on the sample not being detected by SEM/EDS due to the setting of the instrument
- The presence of excessive debris, blood, or soil on the sample masking any gunshot residue particles on the sample

The report can also consider one or more caveats:

- GSR from contaminated personnel can potentially be transferred to people and surfaces with which they come into contact.⁶³
- GSR collected from a subject, such as a hunter, reloader, or firearm user, might be positive due to previous exposure to primer residues.
- GSR is not specific to a particular gun or ammunition.
- It is not usually possible to distinguish GSR particles deposited due to firing from residue deposited by being close to a discharge or through contact with a surface that has GSR on it.⁶⁴

It is recommended that laboratories develop standard operating procedures to address the above GSR reporting considerations. Standard terminology should be used as much as possible in reporting cases, although circumstances can dictate that the terminology needs to be modified to accurately portray the evidence in a clear, concise, and unbiased manner.

REPORT WRITING

REPORT CONTENT

Laboratory reports must contain the following information.

- Unique identifying laboratory number
- Identification of the laboratory issuing the report
- Report date
- Identification of requesting/submitting agency, or person
- An itemized list describing the evidence analyzed
- Results/conclusions
- A description of the technique used
- Name and signature of the author(s)



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Reports should include the following information

- Date and manner of submission
- Requesting/submitting agency's unique identifier
- Disposition of evidence

Results/conclusions:

There is no universal reporting format; however, it must contain results/conclusions. The results must be scientifically accurate and should be written in terms understandable to a layperson. The results/conclusions typically state whether or not GSR was present on the sample(s).

Example: Two particles containing lead, antimony, and barium were found on the tabs from John Doe. Such particles are residue from a detonated primer of a discharged firearm. Particles containing barium/antimony, lead/barium, or lead/antimony were found on the tabs from John Doe. Such particles are found in primer residue, but also may originate from other sources

The criteria used to define GSR (e.g. elemental composition and morphology) must be included in the report.

Example: The sampling devices were examined by scanning electron microscopy energy dispersive x-ray spectrometry and analyzed for elemental composition and morphology of GSR particles.

If gunshot residue is reported:

- The word "unique" is excluded from use in the description of GSR particles.
- A threshold level, if being used in the interpretation, must be specified.

Some laboratories may use a scientifically established threshold level for reporting. If the number of GSR particles does not meet the established threshold level, then those particles must be reported.

- When reporting two component particles, it must be stated that these may have originated from a firearm discharge or non-firearms related sources.

Example: Particles containing barium/antimony, lead/barium, or lead/antimony were found on the lifts from John Doe. Such particles are



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found in primer residue, but also may originate from other sources

- Conclusions drawn from the identification of GSR on a sample from a person must include wording clarifying that the person discharged a firearm, was in the vicinity of a firearm discharge, or came in contact with something that had GSR on it.

Example: Primer residue can be deposited on the hands by circumstances such as firing a weapon, handling a weapon, being in the proximity of the discharge of a weapon or coming into contact with an object that has primer residue on it. The examination itself cannot determine the relative likelihood of these listed circumstances.

- Conclusions drawn from the identification of GSR on a sample from an inanimate object must address the potential that at some time in the history of the item it was in the vicinity of a firearm when it was discharged or came in contact with something that had GSR on it.

Example: The presence of primer residue on an item is consistent with that item having been in the vicinity of a firearm when it was discharged or having come in contact with primer residue on another item.

- If GSR is found on the negative control, this can be disclosed within the report.

If gunshot residue results are negative:

- A statement can be included indicating that a negative result could occur even if the subject discharged a firearm, was in the vicinity of a firearm when it was discharged, or came in contact with something with GSR on it.

Example: The absence of gunshot residue on a person's hands does not eliminate that person from having discharged a firearm.

Example: The absence of primer residue on the hands is consistent with an individual not having fired a weapon. A negative result could also occur from circumstances such as washing the hands, wiping the hands, wearing gloves, sweating profusely, environmental factors including wind and rain, bloody hands, excessive debris on the sample, normal physical activity within 4 to 6 hours passing between firing and sampling, or the weapon not producing primer residue on the hands when discharged.



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- If comparison to known ammunition is conducted, a report should include the details of the elements and combinations of elements that are being considered in making a conclusion. Unless the ammunition in this case is manufactured for a unique application such as ammunition with taggant elements, it should also be noted that these particle types could originate from other ammunition as well.
Example: An unusual proportion of antimony/lead particles was found on the stubs from John Doe; therefore a shell casing, item #10, was analyzed for comparison. The elemental composition of the primer residue particles on the stubs from John Doe, item #2, corresponded to the elemental composition of the primer residue particles from the shell casing, item #10. This proportion, however, could be found in other ammunition.

- An explanatory page may accompany the report if the analyst feels it will assist the reader in understanding the information provided in the report. This may include information regarding formation, deposition, retention of GSR, and scenarios that explain positive, negative, and inconclusive results. It may also include discussion regarding other sources of similar particles (primed explosives devices, fireworks, and brakes).



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APPENDIX A: CONTAMINATION & SAMPLING CONSIDERATIONS

Overview

Contamination, for purposes of this section, refers to the addition of extraneous GSR particles onto a person or object to be sampled or subsequently onto a sample to be tested for GSR. What follows are guidelines for:

A. Procedures for the Prevention of GSR Contamination to a Surface Prior, During, and After Sample Collection.

B. Procedures for the Prevention of GSR Contamination in the Laboratory

An understanding and knowledge of these issues allow the forensic expert to carry out a proper interpretation of the analytical results.⁶⁵ Refer to the Interpretation and Report Writing sections of this Guide.

Understanding the risk of any potential contamination of samples collected for GSR requires some knowledge of the nature and composition of these particles. The particles produced by a firearm's discharge are microscopic in size. Simple precautions such as washing and cleaning can significantly help to reduce the risk of cross-contamination.⁶⁶

A model for the prevention of contamination is that of "Universal Precautions" - measures designed to prevent transmission of pathogens between patients and health care providers. The underlying principle as applied to GSR is to isolate the collector from the surface and the surface from the collector. Being aware of and minimizing the introduction of extraneous particles before, during, and after collection can be accomplished from this perspective.

Another analogy for transfer of material might be to think of chalk dust. It exists in the area of the chalk board and on its accessories. Once contacted, some of the dust particles can be transferred to the new surface.^{67 68 69 70} As activity continues, they will progressively diminish in amount.⁷¹

GSR evidence must be collected in such a way to avoid sample contamination in order to obtain an accurate measure of what is genuinely on the surface being sampled. This can best be achieved by taking appropriate steps at all stages in the process to avoid transfer of unwanted material and by designing procedures that minimize the opportunity, and so the risk, of transfer of material.



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At times it may not be possible to work in a GSR-free environment but by aiming to keep the background levels to a minimum and optimising sampling and handling procedures, the risk of obtaining a false result will be significantly reduced. In addition the reporting scientist should have sufficient awareness of these processes and enough experience to interpret and assess the strength of the findings in relation to these factors.

To assure that the forensic scientists doing GSR analyses reach reliable conclusions they should have:

- A basic awareness of all evidence types and the issues surrounding them, including an appreciation of how sampling for one type of evidence impacts the recovery of another
- Appropriate procedures for Police and Scene of Crime and Laboratory personnel including the use of Personnel Protective Equipment (PPE) and rigorous procedures for cleaning, exhibit and sample handling and packaging using acceptable methods of analysis
- Suitable training and information such as studies concerning occupational contamination.⁷²

Laboratories should establish anti-contamination and decontamination procedures. These procedures should take into consideration that more than one evidence type may need to be collected from the same human subject or evidence item. Protocols should be established to ensure that relevant experts are consulted where potential conflicts arise in the collection of trace evidence samples.

The following guidelines cover best practice procedures for the initial sample collection of GSR at the scene or in the laboratory and ways to avoid any post collection sample contamination that might occur during processing at the laboratory prior to, during, or after analysis.

A. Procedures for Sampling and the Prevention of GSR Contamination at Scenes Prior, During, and after Collection

Due to the nature of GSR, the following guidelines will aid in preventing or mitigating potential contamination of samples collected for analysis.⁷³ All personnel involved in processing the scene and evidence collection are responsible for preventing contamination of any samples collected.

It is acknowledged that not all situations are ideal, and these guidelines do not override issues concerning the safety of the police, the subject, the collector, or members of the public. There will be circumstances in which the collector/examiner has to decide on a case by case basis



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which sequence to collect the various evidence types, as one examination can affect the recovery of another. The collector/examiner must take into consideration what is likely to be of most value to the investigation before proceeding.

Considerations for the Prevention of Potential GSR Contamination during Sampling and Storage

The following protocols should be carried out by the collector and incorporated into the Instructions and Collection Data Sheet developed by Laboratories for inclusion with GSR kits:

- All equipment to be used for the collection of trace evidence, including GSR, should be stored in a clean environment isolated from potential contamination. When undertaking an examination, the equipment (writing materials, sampling equipment, cases et cetera) should be protected from exposure to potential contamination.
- Carry out an initial scene assessment. Note that it may be necessary to don PPE prior to this assessment.
- Avoid dealing with other items or evidence that can be heavily contaminated such as guns, spent cartridges, and other firearms-related items before sampling for GSR.⁷⁴
- Identify a collection area that is isolated from areas that may be contaminated.^{75 76 77}
- Clean the collection area (cleaning protocols should be established by the laboratory)
- Wash hands and put on any PPE before sampling (the minimum PPE used for the collection of GSR is fresh disposable gloves. Minimize or avoid direct contact with the areas to be sampled (e.g. hands of a human subject, clothing or other object) prior to collection.
- Only open one sampling container at a time.
- Take samples as soon as possible to avoid loss of possible evidence.^{78 79}
- Properly seal and label the sample tube to preserve the integrity and continuity of the sample.
- When the sampling has been completed remove and discard the gloves appropriately.
- Fill out the collection data sheet after the samples have been taken.
- Document any deviations from the instructions and checklist provided.



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- Note any other information that could be relevant on the kit data / information sheet.
- Seal the samples and keep the data sheet separate from samples in case it has become contaminated: consider double bagging of the evidence kits as an extra anti-contamination precaution.
- Store the evidence (used) kits in a clean area to avoid contaminating the surface of the packaging: isolate the kits from firearms and ammunition.
- Consider using separate lockers dedicated to GSR Kits and other items for GSR analysis.
- Monitor storage areas to show cleaning and procedures are effective.⁸⁰

Best Practices for Sampling and Mitigation of Contamination

Samples should be collected as soon as possible following the sampling protocols included with the individual kit.

The collector must be aware of cross contamination possibilities and use the proper procedures to mitigate this situation as well as other evidentiary concerns and collection needs.⁸¹

PPE used in the collection of trace evidence, such as GSR, should be lint and particle free. Disposable gloves should be changed between sampling different subjects or objects as needed to avoid transfer between items. Only one sample device should be opened, sampled, labelled and recapped before another is started. Marking pens used for labelling should be cleaned before use and again on completion of the sampling.

Contact with any item to be sampled and any handling of it should be limited as much as possible before sampling. The workspace must be decontaminated and cleaned.

Sampling from Human Subjects

The subject's use of his hands should be minimized before collection. Wherever possible:

- Sample from the subject prior to handcuffing.⁸²
- Subject should be under visual observation before sample collection.
- Do not allow subject to wash/wipe hands.



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- Do not allow subject to use the bathroom before collection. If the urge is irresistible and unavoidable, the subject should be supervised to ensure that he does not wash his hands.
- Do not allow subject to place hands in pockets.⁸³
- Do not remove subjects clothing before GSR sample collection.
- Do not fingerprint the subject before GSR collection.

Samples may be taken from a variety of positions on a human subject; however, in the majority of cases, samples are taken from the subjects hands. Agencies typically will have two or four collections stubs in the GSR kit to sample the following areas of hand with separate stubs for each hand:

- (1) The back of each hand including the thumb-forefinger web as well as all digits and
- (2) The palm of each hand including web of hand as well as all digits.

In addition, samples can be taken from the forehead, cheeks and neck using adhesive sampling stubs. Other exposed body parts, such as the wrists and forearms can also be sampled if the subject is suspected of having washed their hands after the incident.

Moderate pressure of the collection stub surface is applied against the surface to be sampled by repeatedly dabbing. A minimum of 20-30 dabs should be applied to the surface to be sampled using overlapping dabs.

Avoid noticeable deposits of biological materials or other debris that can conceal or obscure GSR particles, such as blood, grease, and dirt. Sampling of moist, sweaty hands cannot always be avoided, but the drier and cleaner areas should be done first.

- Considerations when sampling head, facial and nose hair: Adhesive stubs may also be effective in sampling from hair.^{84;85} Alternate sampling devices including a comb fitted with a mat of non-woven fibers and vacuum filtration have been employed for such samples.
 - Samples have been taken from inside the nose using cotton tipped swabs; however, these require considerable sample preparation to mount the sample for analysis by SEM/EDS.
- Several stubs may have to be used to sample the entire area effectively. The collector's documentation should also clearly identify each sample and reference the location sampled.



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Sample Collection from Clothing, Vehicles and Other Objects

As with samples taken from human subjects, samples taken from clothing, vehicles and other objects should be subject to procedures based on the above recommended protocols (refer to “Considerations for the Prevention of Potential GSR Contamination for Sampling and Storage”). In some cases the procedures for GSR collection may also be subject to protocols for the collection and/or testing of other evidence types, such as fingerprints and DNA.

Sampling procedures will be dictated by incident/scene circumstances.

Avoid noticeable deposits of biological materials or other debris that can conceal or obscure GSR particles, such as blood, grease, and dirt. The number of collection stubs used is dictated by how quickly the stub surface is loaded with sample and loses its adhesiveness. Wet surfaces should be allowed to dry before sampling. The surface to be sampled must be protected from potential contamination while it dries and evidence must be secured. Clothing, for example may be dried in lockable drying cabinets with sub-micron HEPA filters that can be de-contaminated before and after drying.

These guidelines do not pertain to the estimation of the muzzle to bullet hole/shot damage distance. If this is a consideration in the investigation, an appropriate sampling and testing regime will have to be established.

★ **Sampling from Clothing**

When possible, clothing is sampled in situ at the scene to minimize the loss of GSR as a result of activity in removal from the subject (living or dead), transport, and packaging.⁸⁶

If clothing has to be removed from a living subject for subsequent GSR (and possibly other trace evidence) sampling in the laboratory, it is recommended that the subject be directed to remove the garment himself/herself and place it, with minimal disturbance, into a large paper evidence bag. The bag may be held open to assist in the process by a collector who has previously washed his/her hands and is wearing fresh, disposable, gloves (as a minimum PPE requirement).

If sampling of a deceased victim’s clothing is required it is preferable that the clothing is packaged at the scene as blood seeping from wounds may obscure GSR deposits and other evidence such as blood spatter patterns.

It is recommended that the garments are cut into two sections: the uppermost/exposed sections of each garment that can be lifted away from the body; and the section underneath the body that is left behind once the body has been lifted away. Each section of each garment should be packaged separately, preferably without folding. If any part is blood soaked (with wet blood), paper can be placed in a wad over the stained area so that blood does not transfer to unstained areas.



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The area to be sampled is dictated by what areas were most likely to have been exposed to the plume of the discharge. This information may be available from the investigators. In any case, collection stubs should be clearly labeled as to what areas were sampled. The number of stubs used will depend upon the area to be sampled and how quickly the stubs are filled; i.e., no longer has adhesiveness

The most likely areas to sample include;

- Right and left cuffs and wrists of long sleeved shirts or jackets
- Right and left shoulders if a long gun was used
- Pockets or back waistband- if there is reason to believe that is where the gun was carried
- Headwear including the underneath side of hat visors
- Gloves (sampled like hands)

★ ***Sampling from Other Fabric Objects***

Cloth furnishings and bedding should preferably be sampled at the scene as the maximum investigative context can be established. Smaller, unfixated fabrics such as towels may be packaged and forwarded to the laboratory for sampling.

★ ***Sampling from Vehicles***

Vehicles may be involved in a number of ways in shooting incidents. The most common of these are:

- Shots fired from a vehicle (drive-by shooting)
- Shots fired inside a vehicle
- Shots fired adjacent to or over a vehicle
- A vehicle used by the shooter to leave the scene of crime

Each scenario will present different sampling requirements.

Where shots are believed to have been fired from a vehicle, samples can be taken from the exterior and interior surfaces around the window(s), although it is believed that air velocities and hard smooth surfaces will hinder the deposit of GSR. Other locations where the firearm may have been placed after the shooting may also be sampled.



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When a firearm is discharged inside a vehicle, GSR is usually found on most surfaces inside the vehicle. In the case of shots being fired adjacent to or over the vehicle, generally sampling will be limited to the exterior panels and windows.

Where a shooter decamps from a scene of crime in a vehicle, likely contact areas within the vehicle, such as the steering wheel, gearshift lever, and door handles can be sampled.

The exterior surfaces of most vehicles are smooth and impervious. Consequently, they will not retain gunshot residue deposits unless there are other materials adhering to the surface of the vehicle that facilitate the persistence of particles. If such samples are required, the exterior surfaces of the vehicle should be sampled as soon as possible at the scene.

Sampling from the interior of a vehicle may require that the sample collector enter the passenger or driver's compartment. This entry should be kept to a minimum and with the collector wearing full PPE.

If the vehicle is also to be sampled for fingerprints or DNA (and possibly other traces), appropriate precautions must be taken to eliminate the possibility of contamination and loss of, all evidence. Furthermore, the GSR sampling should not result in the obliteration of fingerprints or removal of DNA traces, if possible. Avoid smooth impervious surfaces to prevent damage or removal of latent fingerprints inside a vehicle, however, trace DNA may be sampled from all surfaces on which GSR may also be found. Consequently, a strategy of sampling for each will have to be established through discussion with the investigators and biological examiners.

The inside of the vehicle should be sampled before contents are inventoried and removed. The order of sampling and fingerprinting will vary depending on the case circumstances, however, in most cases GSR sampling will precede fingerprinting followed by DNA sampling and finally other trace evidence forms, such as glass and fibers. **Note:** GSR sample stubs may be examined microscopically for the presence of glass fragments, fibers of interest or other relevant physical evidence materials prior to SEM/EDS analysis.

★ **Sampling from Interior and Exterior Structures**

Miscellaneous types of surfaces can be sampled to obtain information about their proximity to the discharge of a firearm; to establish whether a person with GSR on them may have contacted a surface; or whether an area of damage was caused by a bullet or shot. These may include phones, door knobs, handles, etc.

Though SEM/EDS examination of GSR does not yield an estimate of the distance from muzzle to target, a representative sample(s) of areas could reflect the item's history in relation to being exposed to GSR.



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The Collection Information Sheet should be filled out after sample collection and included along with the other submission paperwork. Avoid contamination of the Collection Information Sheet and the other paperwork by using non-contaminated, new material for packaging of evidence.

Failure to follow the above guidelines could result in compromising and/or loss of GSR evidence.

B. Procedures for the Elimination of GSR Contamination in the Laboratory

Contamination could potentially occur at several points in the processing of items or samples for GSR unless appropriate procedures and precautions are in place. The length of time samples are exposed when handling and preparing them for analysis will generally dictate the level of contamination risk. Good laboratory practice will result in minimal or negligible risk that any GSR will get onto a sample inadvertently while in the laboratory.

While the suggestions in this section of the guide are all good laboratory practices, data from a recent SWGSR contamination study indicates that, if the samples are handled carefully, the chance of contaminating the surface of the sampling device by GSR particles that can be on the outer surface of a collection vial is extremely remote. In these tests no GSR particles were found on (unused) stubs from sampling devices that were covered with GSR immediately after firing various types of weapons and ammunition.⁸⁷

The following factors have been identified as areas for consideration that will help to provide a GSR-free environment in which the work can be carried out. Some or all of the following can be applicable:

- The physical separation of SEM laboratory and GSR trace sampling room(s) from a firearms section or firing range
- Separation of sampling areas for: items that are likely to be heavily contaminated with GSR (fired cartridge cases, firearms, victim's clothing, etc.); and items likely to have less GSR on them, such as suspect's clothing.
- Access to the SEM laboratory and GSR sampling room(s) restricted to staff who work in the area. Enforceable protocols should be established that identify "no-go" areas and common areas. The protocols should take into account all personnel that may access any given area, including scientific and technical staff, visitors, police, janitors, maintenance engineers and administrative personnel.



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- Procedures for personal decontamination if GSR personnel are required to enter an area of high contamination then return to the SEM/GSR restricted area.
- Protocols for the use of PPE:
 - In sampling and examination rooms. The protocols should describe when to change PPE during and between examinations of multiple exhibits. Consideration should be given to changing gloves after physically handling an exhibit and before handling a camera, writing implements or examination equipment.
 - In the sample preparation and SEM room(s).
- Protocols for de-contamination and cleaning of the sampling room(s):
 - Prior to an examination.
 - Between examinations of exhibits from the same subject (i.e. different items of clothing worn by the same person).
 - Between examinations of exhibits from different persons.
 - On the completion of sampling.

The protocols may need to take into account other evidence considerations, such as DNA contamination.

Consideration should also be given to including the maintenance of a log of examinations and cleaning.

- The potential of examination notes and case files as a means of transferring contamination.
- Protocols for cleaning the SEM and sample preparation room(s).
- The placement of sticky mats at strategic locations to restrict the movement of GSR particles around the laboratory
- Protocols for the receipt and storage of evidence. These should include the following:
 - All exhibits accepted for examination must be under evidence seal on receipt.
 - The evidence seal should render the packaging impermeable to ensure that GSR particles cannot pass from or into the package.



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- Firearms and fired cartridge cases must be stored separate from exhibits received for GSR examination.

Recommended Practices to Monitor GSR Levels

Regular monitoring of the laboratory environment can help to show if the precautions being taken and the cleaning routines are effective. Suitable areas to be monitored include the GSR kit preparation area, SEM and sample preparation area, bench tops used for bulk item sampling, storage areas, etc. Background surveys can also be used as a means of identifying potential areas of concern and to provide reassurance that such situations have been considered.^{88 89}

The following processes may be used to evaluate and identify contamination sources in the laboratory:

- Monitor work areas regularly: the use of Control samples taken from a prepared work area prior to commencing an examination provides reassurance that the area was free of contamination for a particular case.
- Processing an unused sample stub at the same time as casework samples so that it passes through all of the handling, packaging, sample preparation and analysis steps as the casework samples will demonstrate extraneous material has not been introduced within the period of testing.

This type of sampling may also be used as a “blind-negative” proficiency test.
- Known blank samples placed in the SEM chamber and or the carbon coater can be periodically analyzed for GSR particles. This demonstrates that particles remain fastened to the adhesive tape samples and do not become dislodged during coating or analysis.
- Sampling from non-restricted areas of the laboratory in which GSR personnel may be exposed to contamination. Suggested areas include common rooms and evidence management (receipt, storage and allocation) areas.
- Random sampling of visiting personnel and personnel submitting evidence. These samples will assist in identifying the risk of non-laboratory personnel introducing contamination into the laboratory.

The scope and regularity of testing within the laboratory will depend on many factors, including laboratory design and extent of separation of critical areas pertaining to GSR and firearms, laboratory protocols pertaining to access of personnel, etc.



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Recommended Practices if GSR Contamination is Detected

Monitoring for contamination within the laboratory operates on two levels:

- Identifying a potential risk in processing GSR casework
- Identifying potential risks within the general laboratory environment.

When contamination is detected on Control samples used to monitor the processing of GSR case work, case work may have been compromised. However, when it is detected on samples used to monitor non-GSR common laboratory areas and non-GSR personnel, it does not have a clear and direct link to casework. Consequently, the management of a contamination issue will be different depending on which process the contamination pertains to.

The laboratory should have a procedure in place in the event that contamination is detected on any monitor sample that should include:

- Diagnosis of the contamination incident
- A review of the impact on the relevant case, if applicable.
- Identification of other cases that might be affected and review of each relevant case.
- Remedial action required if the contamination is believed to have compromised case work. This will generally include informing the appropriate authorities and withdrawing the report (if one has already been issued).
- A set of actions to rectify the problem. The procedures involved will depend on the nature of the contamination and its impact on casework.

Where the contamination is detected in a case work Control sample:

- The areas indicated by the contamination should be thoroughly cleaned and tested for residual contamination.
- Case work can resume once the contamination procedures have been demonstrated to have been effective.

Where contamination is detected in other areas of the laboratory or on non-GSR personnel the risk to GSR case work should be comprehensively assessed. As a consequence of this assessment, it may be necessary to modify laboratory procedures and protocols, including:

- Expanding the scope and regularity of this form of monitoring.
- Increasing the restricted access zones.



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- Introducing new facilities to control the passage of particulate material into critical areas.
- Establishing restrictions on the carrying of firearms into certain (or any, if feasible) areas of the laboratory by attending police members (or laboratory staff, if they are police members issued with a firearm).
- Establishing separate areas in common rooms and catering facilities for GSR and Firearms personnel.
- Establishing more regular and thorough cleaning of common rooms and other areas in which staff may interact.
- Introducing more rigorous personal decontamination and PPE procedures for GSR staff prior to entry into the restricted zone.

Summary of Best Practices in the Laboratory

Facilities

Work areas used for GSR work:

- Should be designed or situated to be free of GSR particles:
- Should not be located close to firing ranges or where firearms, ammunition, or associated accessories are examined.
- Should not be connected to the same air conditioning or ventilation system as areas that may be a source of GSR.
- Should be regularly cleaned and monitored for the presence of any GSR.

Equipment and other surfaces in the GSR sampling and SEM rooms should be regularly cleaned. This should include:

- Bench tops.
- Consoles, keyboards and monitors.
- The interior of the SEM chamber.
- The interior of the carbon coater chamber.
- Sample holders.
- Any instruments used to handle or prepare samples.

Samples should be kept in closed containers, and the time they are exposed kept to a minimum.



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Access

Access to key areas such as sampling areas, sample preparation areas, and analysis rooms should be limited to authorized personnel.

Regular firearms users or persons carrying firearms should not be allowed to enter the GSR facilities. Similarly, GSR personnel should not enter the firearms facilities unless it is unavoidable.

Where any personnel that may have been exposed to GSR needs to enter the GSR facilities on the same day as being exposed, they should:

- Change their outer clothing.
- Put on the required PPE before entering the GSR facilities.
- The persons clothing that had been exposed to GSR must be laundered before it is worn in the GSR facilities.

Tours of GSR analysis area should be kept to an absolute minimum if they cannot be avoided all together. If they are permitted, tours of the GSR facilities must precede any tours of the firearms facilities.

Similarly, personnel visiting the GSR facilities must not have entered the firearms facilities previously.

A record of visitors can assist with investigating any contamination that may subsequently be found.

Many of these issues can be dealt with by use of a dust free “clean room” or by working in conditions suitable for DNA examinations.

Personnel

Staff involved in GSR examinations should not themselves be regular firearms users or carry a firearm. When exposed to potential contamination, staff need to take steps to mitigate this issue (refer to “Access”, above). This should involve washing hands, wearing protective clothing when collecting samples, or changing outer garments before entering the GSR analysis area,

Procedures

★ *Receipt of Items*

It should be assumed that the exterior packaging of items submitted for GSR analysis and examination could be contaminated, depending on how they have been stored, transported, or handled.



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★ **Storage of Items**

GSR exhibits should be stored separately from firearms and ammunition. The storage areas for the GSR kits can be periodically cleaned.

★ **Examination of Items and Sample Preparation**

To avoid contaminating the contents when an item is opened, the surface of the packaging can first be wiped with a damp towel. Protective clothing such as gloves and a laboratory coat should be worn for examining clothing and other objects. Measures necessary to preserve other evidence types, such as DNA, should be taken.

- **Kit samples**

The samples in kits are protected inside individual sample containers and do not need to be handled directly. Equipment such as forceps should be cleaned with a wet wipe or tissue between items.

- **Clothing**

Sample collection depends on the item(s) to be sampled; size, condition, prior handling, etc. Additional factors that can influence the number of samples collected include: the known case circumstances of the incident, such as the time since seizure of the item and the event; the requirements of the investigation; and possible limitations, such as retention of GSR from previous, non-related firearm discharges by the wearer.^{90,91 92}

- **Vehicles**

All personnel, including the sample collector, should remain outside of the vehicle if possible. Precautions need to be taken to prevent any possibility of cross contamination as described in “Sampling from Vehicles”, above.

★ **Analysis**

The goal is to have a sample arrive at the SEM for analysis with only material originating from the sample on it using the above suggestions and recommendations and by having laboratory procedures and protocols that are proven, documented, and routinely performed to prevent contamination. The next step is to apply a similar strategy during the analysis and post analysis to continue to ensure that no contamination occurs to the sample.

The main considerations in the preparation of samples and subsequent SEM analysis are:

- Labelling or tracking each sample stub prior to carbon coating in such a way that samples can be correctly identified.
- Handling procedures for the sample stubs to minimize any removal or transfer of material from or onto the sample surface by using clean forceps and wearing gloves if necessary.



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- Loading the specimens onto the SEM stage must be accomplished with the same careful handling as that employed in carbon coating.
- Removal of the specimens from the SEM stage and placing them back into their correct containers must also be performed with care. The SEM sample stage can be wiped between runs using a moist cloth or lint-free tissue. Once the samples have been removed pressurized air may also be used to remove any dust from the chamber and seals.
- The opening of kits, as well as the samples within, can be done on a bench top remote from the SEM or carbon coater. In other words, samples are individually loaded from an area that is not in close proximity to the SEM or the carbon coater. If not possible, the work space adjacent to the equipment should be thoroughly cleaned between processing jobs.



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APPENDIX B AMMUNITION

The basic components of a cartridge include the primer, cartridge case, propellant, and projectile(s). Other materials incorporated into the ammunition can include coatings (paint, laminates, etc), lubricants, and polymers.

The function of the primer is to initiate the explosive combustion of the propellant through a shock sensitive reaction caused by the impact of the firing pin of the firearm. There are two basic primer mechanisms used in modern ammunition: rimfire cartridges, employed mainly in .22 caliber ammunition, and centerfire cartridges, employed in all other small arm (revolvers, pistols, rifles, etc.) ammunition.

In rimfire ammunition, the shock-sensitive primer mixture is applied into a flared cavity around the base of the cartridge case. On discharge, the firing pin strikes the rim of the cartridge case compressing the flared rim of the cartridge case.

Centerfire primers are made of three basic components: the cup, anvil, and priming mixture. The primer cup, which contains the primer mixture, is inserted into a circular cavity in the center of the face of the cartridge case. Two type of cartridge design, Berdan and Boxer, are used in small arms ammunition. These differ in their anvil design. In the Berdan type (named after its inventor, the American engineer and military officer Hiram Berdan), the anvil is an integral part of the cartridge case and protrudes down into the primer cup. In a Boxer cartridge (named after its inventor, Edward M. Boxer of the British Army), the anvil is a separate metal component that is inserted into the primer cup. Berdan cartridges are used mainly by former Eastern Bloc and Asian manufacturers and Boxer cartridges are used by most European and American manufacturers. A third type of centerfire cartridge is the Battery cup, used mainly in shotshells.

A metal, plastic, or paper disc is often placed over the priming mixture inside the primer cup. These discs provide a physical barrier to hold the priming mixture in the cup during transport and protect it. Paper discs are mainly resin coated. In some instances they serve to deposit anti-corrosive material in the barrel. Tin foil is also used for this purpose in some ammunition, particularly from eastern Europe.

Primer cups are generally made of brass (70/30 cartridge brass - Cu/Zn). Often they are coated with nickel. Although less common, some cups are made of aluminum as found in some CCI Blazer® cups.

In Boxer cartridges, the anvil is often made of brass, usually 70/30 cartridge brass.

Cartridge cases are predominantly made of cartridge brass. This is a 70% copper and 30% zinc alloy. Some cases are composed of aluminum, stainless steel (Fe, Ni, and Cr), and polymer coated steel. In some instances, steel cases are “washed” or electroplated with copper or nickel to prevent corrosion and provide an aesthetic finish. Some .22 caliber ammunitions have cobalt-plated cases.



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Projectiles include bullets, pellets and slugs, mainly made of lead or incorporating a lead core. Bullets can be unjacketed, semi-jacketed, or jacketed.

Unjacketed bullets are often encountered in lower caliber firearms. Lead bullets may be hardened with antimony, tin, or both. .22 caliber bullets may also be copper coated or “washed” (this is not considered to be jacketed). In this process, the lead surface is enriched with antimony prior to the application of the copper coating. In rare cases, metal coatings have been applied to large caliber bullets and shot. Zinc coatings have been applied to wad-cutter projectiles used for practice ammunition.

Jacketed and semi-jacketed bullets are often encountered in higher caliber type firearms. Often, the jacket material is harder than the core material with the exception of armor piercing bullets. Compositions include these:

- Cu 90-95% with Zn (most common, also called “gilding” metal) alone or coated on brass or steel
- Steel plated with Ni or Cr
- Steel plated with Cu and finally Sn (RUAG/MEN; still used for police training in Bavaria)
- Bronze (Cu, Sn), Cu, or Al coated brass
- Cu, Bronze, or Al alone

In some applications, jackets are modified to incorporate a tip that can include Pb, Al, steel, or a synthetic polymer.

Bullet core components include these:

- Pb or Pb hardened with Sb (most common)
- Pb hardened with Sn, Cu, Zn, Fe, or W alone
- Stainless Steel (Fe, Cr, Ni)
- Brass (Cu, Zn)

Armor-piercing bullets are usually jacketed with a metal that is softer than the core and often has a tip filled with Pb. This prevents the penetrator from shattering upon impact. The core is usually steel hardened with W, Cr/V, Cr/Mo, Mn/Mo, W/C, or W/Cr.

Tungsten carbide is also used as a core material and can contain low amounts of Ti, Ni, and Fe.

Depleted uranium is also used in some armor-piercing ammunition predominantly encountered in military applications with larger caliber weapons and firearms.

Shotgun pellets and slugs are generally made of Pb or Pb hardened with Sb. Steel pellets are also encountered. In some instances the steel pellets can be plated with Ni or Cu.



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Firearm chemistry has changed significantly over the last two centuries and in particular over the past 60 years. Consequently, a variety of primer and propellant types from across this period of manufacture could be encountered in forensic examinations (refer to *History of the Development of Firearms Chemistry*, below).

Firearms

The firearm itself can contribute to the elemental compositions seen in gunshot residue. The contributions originate mostly from the barrel and the cylinder in revolvers. Most barrels are made of carbon steel and stainless steel and are sometimes coated with other metals or alloys to provide protection or in projectile activity. Titanium has also been used in some revolvers.

Gun bluing is used for providing a protection from corrosion. Modern methods involve electrochemical, hot and cold processes at the time on manufacture or by application using aftermarket gun bluing kits. These processes are based upon an oxidation process which is used to develop a protective surface coating. Generally, the techniques may use some form of chromium, selenium, copper or other metals.

Chromium and molybdenum alloy steels are also available in barrels and can contribute to the composition of gunshot residue. It should be noted that manganese is also present.

History of the Development of Firearms Chemistry

The first known use of a chemical substance, or mixture, for firearm ignition is by the Reverend Alexander Forsyth, who studied a group of chemical compounds called metallic fulminates whose existence had been known from around 1800. It was also known that they exploded with a flash when struck a sharp blow with a hard object. In 1805, Forsyth applied this property of metallic fulminates to firearms ignition, thereby inventing the percussion system of ignition. In 1807, he took out a patent on his invention by which a pivoted magazine deposited a few grains at a time of the priming mixture (probably mercury fulminate) into a touchhole in the barrel of the firearm. The mercury fulminate was detonated by a blow from the hammer of the firearm, sending flame through the touchhole to ignite the propellant, achieving “instant” ignition. This led to several short-lived innovations including the tubelock, patchprimers, and the pill-lock eventually leading to the percussion cap, which proved to be the most efficient and practical way to package the primer. The development of the percussion cap (small waterproof copper cups) is credited to Joshua Shaw in 1816. The cap (primer) was placed over a permanent hollow nipple, screwed into a flash hole in the gun barrel, and detonated by the crushing impact of the hammer.

At that time, black powder was the only form of propellant in use. Black powder or gunpowder is a mixture of potassium nitrate, sulfur and charcoal. It is still used today, predominantly in modern muzzle loading firearms. Black powder substitutes are more commonly used for this type of firearm as they are less sensitive and provide a more efficient burn. Some common substitutes include Pyrodex®, Triple Seven®, and Clear Shot®. Generally, they contain charcoal, graphite, sulfur, potassium nitrate, potassium perchlorate, cyanoguanidine and sodium benzoate.



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The earliest cartridge design still in use today is the rimfire cartridge. The rimfire cartridge was patented by French gunsmith Houllier in 1846 and developed by another French gunsmith, Flobert. They are used today mostly in 0.22" ammunition.

Centerfire cartridges were produced by Pauly in 1808, but it was not until 1854 that the Smith & Wesson perfected and patented both the centerfire and rimfire metallic cartridge case.

Early priming compositions consisted of mercury fulminate and potassium chlorate, along with other ingredients. With the introduction of metallic cartridge cases about 1850, it was found that brass cartridge cases were unsuitable for use with priming compositions containing mercury fulminate as the brass was embrittled due to mercury amalgamation of the zinc. This made the spent cartridge case useless for reloading purposes, and reloading was essential for economic reasons. Initially the use of copper cartridge cases solved this problem. In 1869, Hobbs, by the use of internal varnishing of brass primer cups and brass cartridge cases, made the use of brass and mercury fulminate possible by preventing the direct contact of the brass surface with the primer mix.

Whenever black powder was used as a propellant, a large amount of fouling was deposited on the inside of the barrel. On combustion, black powder produces about 45% of its original weight as hot gases and 55% as solid residues in the form of dense white smoke.

Smokeless powders were introduced between approximately 1870 and 1890. The first smokeless powders were produced through the nitration of cellulose materials such as cotton. Hence, the original name of "gun cotton".

Modern smokeless gunpowders are grouped into three basic categories: single, double, and triple-base. Single-base powders consist mainly of nitrocellulose (NC). A powder containing nitroglycerin (NG) in addition to NC is classified as double-base. Triple-base powders contain NC, NG, and nitroguanidine salts. Most smokeless powders for small arms are either single- or double-base. Triple-base powders are used in rockets and military ordnance and are seldom encountered in forensic work.

In addition to the major constituents (NC and NG), smokeless powders also contain stabilizers such as diphenylamine, ethyl and methyl centralite; burning modifiers such as 2,4- and 2,6-dinitrotoluenes; plasticizers such as dibutylphthalate; and coating ingredients such as graphite. Different nitrogen oxides formed during the decomposition of NC during storage react with stabilizers (the most common is diphenylamine) to form nitro- and nitroso-compounds. Thus, 2-nitrodiphenylamine, 4-nitrodiphenylamine, and N-nitrosodiphenylamine are commonly found in smokeless powders.

As smokeless powders were harder to ignite than black powder, larger priming loads were necessary. Consequently, higher pressures were experienced with smokeless powders and, as they burn cleaner than black powder, they produced much less fouling. The relatively clean surfaces remaining in the barrel interior after the combustion of smokeless powder became rusted, even when the gun was cleaned immediately after use. This is due to the formation of chloride salts after the combustion of the potassium chlorate containing primers. The heavy



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residue left after the combustion of black powder substantially protected the metal surfaces from the effects of the salt, and to some extent from the effects of metallic mercury released after combustion of the primer. The problems associated with the use of mercury fulminate and potassium chlorate led to a search for suitable alternatives, and the chemical reactions occurring within the cartridge case and the firearm were intensively studied. The objective of the study was to produce a satisfactory priming composition that was both noncorrosive and nonmercuric (NCNM).

The first noncorrosive primer was produced by the German firm of Rheinisch-Westphälische Sprengstoff AG (RWS) in 1891, based on mercury fulminate, barium nitrate, antimony sulfide and picric acid. In the following years several other noncorrosive primers were introduced, all based on mercury fulminate as initiator.

The first practical NCNM primer mixture with satisfactory ignition properties and good shelf life was produced (again) by RWS in 1928. This type of primer was given the general name of "Sinoxid" ("Sinoxide", "Sinoxyd"). This was the forerunner of all modern NCNM priming compositions and had the following general composition:

- Lead styphnate - 25% to 55%
- Barium nitrate - 24% to 25%
- Antimony sulfide - 0% to 10%
- Lead dioxide - 5% to 10%
- Tetracene - 0.5% to 5%
- Calcium silicide - 3% to 15%
- Glass powder - 0% to 5%

With very few exceptions, U.S. commercial primers became noncorrosive about 1931 but because of stringent U.S. government specifications for military ammunitions, which could not be met by the earlier versions of the new NCNM primer mixtures, it was not until the early 1950s that U.S. military ammunition became noncorrosive. This was because early NCNM commercial priming mixtures suffered erratic ignition and unsatisfactory storage stability, and as large quantities of small arms ammunition are stored as a war reserve, military ammunition must have unquestioned reliability and storage stability.

In the UK both commercial and military ammunition used primers that were both mercuric and corrosive until the gradual changeover to NCNM primers that was completed during the mid-1950s and early 1960s.

Primer Composition

The initiating explosives are basically primary high explosives that are so sensitive as to explode when struck by the striker or firing pin of a weapon, starting a flame to ignite the propellant in a small-arms cartridge. Explosives used include azides, fulminates, diazo compounds, nitro or



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nitroso compounds — for example lead or silver azide, mercury fulminate, lead styphnate, TNT, and PETN (which also act as sensitizers).

The explosive ingredient in Sinoxid-type primers is lead styphnate (lead trinitroresorcinate), which is very sensitive to static electricity, and fatalities have resulted from handling the dry salt. Preparation of the pure salt is difficult, and many patented preparations, including basic modifications, exist. Some claim special crystalline forms and/or reduced static electricity hazard. Explosive ingredient substitutes for lead styphnate were sought that would be easier to make and safer to use. These included lead azide, diazodinitrophenol, lead salts of many organic compounds, complex hypophosphite salts, picrate-clathrate inclusion compounds, and pyrophoric metal alloys.

Despite the search for alternatives to lead styphnate and the considerable experimentation with primer compositions, in the UK and the U.S., the vast majority of modern ammunition still contains Sinoxid-type primers with lead styphnate and barium nitrate together typically making up 60% to 80% of the total weight. Mercury fulminate/potassium chlorate-based primer compositions are currently manufactured by some Eastern Bloc countries, although they also manufacture compositions based on lead styphnate.

Oxidizing agents are used in primers to increase the heat of ignition. Oxidizers used include barium nitrate, potassium chlorate, lead dioxide, and lead nitrate.

Fuels used include antimony sulfide (which also acts as a frictionator), gum arabic (which also acts as a binding agent), calcium silicide (which also acts as a frictionator), nitrocellulose, carbon black, lead thiocyanate, and powdered metals such as aluminum, magnesium, zirconium, or their alloys.

Frictionators used include ground glass and aluminum powder (which also acts as a fuel).

Sensitizers used include tetracene, TNT, and PETN.

Binders used include, among others, gum arabic, gum tragacanth, glue, dextrin, sodium alginate, rubber cement, and Karaya gum.

Primer mixtures can be divided into six categories:

- Mercuric and corrosive
 - Mercury fulminate, potassium chlorate, and antimony sulfide based. A typical primer discharge residue particle composition is potassium, chlorine, and antimony (rarely containing mercury).
 - Mercury fulminate, potassium chlorate, and antimony sulfide based, sealed with tin foil. A typical primer discharge residue particle composition is potassium, chlorine, antimony, and tin (rarely containing mercury).
- Mercuric and non-corrosive



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- Mercury fulminate, barium nitrate, and antimony sulfide based. A typical primer discharge particle composition is barium, antimony, and sulfur (rarely containing mercury).
- Mercury fulminate, barium nitrate, and antimony sulfide based, sealed with tinfoil. A typical primer discharge particle composition is barium, antimony, tin, and sulfur (rarely containing mercury).
- Non-mercuric and corrosive
Potassium chlorate, lead thiocyanate, and antimony sulfide based. A typical primer discharge particle composition is lead, antimony, potassium, and chlorine.
- Non-mercuric and non-corrosive (NMNC)
 - Lead styphnate, barium nitrate, and antimony sulfide based (Sinoxid type). A typical primer discharge particle composition is lead, barium, and antimony
 - Sinoxid-type primers, containing aluminum powder as frictionator and fuel. A typical primer discharge particle composition is lead, barium, antimony, and aluminum.
 - Sinoxid-type primers, sealed with tinfoil. A typical primer discharge particle composition is lead, barium, antimony, and tin.
 - Lead styphnate, barium nitrate, and calcium silicide based. A typical primer discharge particle composition is lead, barium, calcium, and silicon.
 - Lead styphnate, barium nitrate, and calcium silicide based, sealed with tinfoil. A typical primer discharge particle composition is lead, barium, calcium, silicon, and tin.
 - Lead styphnate, barium nitrate and glass-based (common to .22 caliber ammunition). Borosilicate and soda-lime glasses are used as frictionators. A typical primer discharge particle composition is lead, barium, and silicon.
 - Lead styphnate, barium nitrate, and lead thiocyanate based (mainly in 0.22" rimfire ammunition types) A typical primer discharge particle composition is lead and barium.
 - Lead styphnate based (e.g. Remington, "U" or "Rem" headstamp, 0.22" rimfire ammunition types). A typical primer discharge particle composition is lead only.
- Miscellaneous
 - Phosphorus-containing Eley (UK) 0.22" rimfire ammunitions. Primer composition is lead styphnate, barium nitrate, and lead thiocyanate based. (The presence of phosphorous is due to the manufacturing process of lead



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stypnate.) A typical primer discharge particle composition is lead, barium, and phosphorous.

- Lead-free/heavy metal free/Clean Ammunition

There is a wide range of formulations used by manufacturers in this class of primer (refer to *Lead-Free/Clean Ammunitions*, below)

Lead-Free/Clean Ammunitions

There are many ammunition brands that incorporate lead-free primers distributed throughout the United States. They include, but are not limited to, the following:

- Winchester Winclean, SuperClean NT, Super Unleaded
- Remington/UMC Leadless
- Federal BallistiClean
- Speer Lawman CleanFire
- CCI Blazer Lead Free
- Springfield .30-06 Pb free Rifle Ammunition
- Hornady Pb free GMX Rifle Ammunition
- Black Hills Gold [Hunting Rifle Ammunition]
- Precision Ammunition [Copper-Matrix NTF] RUAG Green Fire Primer
- Cor-Bon DPX Solid Cu Projectile
- Magtech CBC Clean Range
- DoubleTap Ammunition 416 Taylor 350 Grain Barnes Triple-Shock X-Bullet
- Extreme Shock CT-2 Tactical Ammunition 40 S&W 100 Grain Copper Jacketed Ballistic Tip
- Federal Premium Vital-Shok Ammunition 12 Gauge 2-3/4" 1 oz Barnes Expander Hollow Point Sabot Slug
- Federal Premium Cape-Shok Ammunition 370 Sako Magnum 286 Grain Barnes Triple-Shock X-Bullet
- Winchester Reduced Hazard Shot Shell Primers [Fe (50-55 %), Cu (20-35%), Zn(1-5%) and Bismuth Subnitrate (1-2%)]

GSR originating from US lead-free ammunition listed above may include the following elemental combinations:

- Sb/Ba
- Al/Ba/Si



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- Al/Sr
- Al/Na
- Al/Si/K
- K/Cu/Zn
- K only
- K/Si
- Si/Ca
- Sr/Cu/Zn
- Sr/Al/Si
- Sr only
- Cu/Zn

European lead-free/clean ammunition primer compositions are not quite as extensive as those found in the United States. They include but are not limited to the following:

- Fiocchi (GFL) (Pb-Free, with Sb/Ba or also Al/Si/K/Zr (“ZETAPI” Boxer Primers))
- Dynamit Nobel (RUAG) Sintox (Zn/Ti)
- Metallwerk Elisenhuetten/Nassau (MEN) (Zn/Ti)
- Sellier & Bellot Nontox

GSR originating from the above European lead-free ammunition includes the following elemental combinations:

- Sb/Ba
- Zn/Ti
- Al/Si/K/ (Zr apparently not observed)

The cartridge case, projectile and propellant will also contribute to the composition of GSR originating from lead-free ammunition.

“Tagged” Ammunition

Taggants have been introduced into police ammunition in Germany in order to identify GSR associated with the discharge of police firearms in shooting incidents. In addition, the introduction of these taggants has eliminated the issue of police as a source of GSR contamination from police firearms.

The taggants have been added to either the lead-free primer or the propellant and provide a distinguishing characteristic of GSR sourced from police ammunition.



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For a taggant to be effective, the chosen element must be uncommon, not present in ammunition used by non-law enforcement personnel, available at low-cost, and must not diminish the primer or propellant reactions. Ultimately, gallium and gadolinium were selected and, after extensive testing resulted in two types of ammunition, they were introduced in 2001. The current available ammunition types are:

- Metallwerke Elisenhütte GmbH Nassau, Germany (MEN, subsidiary of MAGTECH/CBC, Brasil) and Polizei-Einsatz-Patrone (PEP)- PEP II (Taggant: Gallium, Ga)
- RUAG Ammotec GmbH, Germany and RUAG Ammotec AG, Switzerland (Headstamp “DAG” - “Dynamit-Nobel Aktien Gesellschaft”, subsidiary of RUAG, since 2002) - RUAG Action 4 (Taggant: Gadolinium, Gd)

MEN PEP II tagged ammunition incorporates:

- A Tombac (copper/zinc) bullet with a blue polyethylene ball-nose containing copper flakes
- A nickel plated (interior and exterior surfaces), brass, primer cup
- A brass boxer cartridge
- A non toxic (Ti, Zn) primer
- Single or double base propellant tagged with gallium (with copper and tin)

RUAG ACTION 4 tagged ammunition incorporates:

- A brass bullet containing a trace of lead with a yellow polyethylene tip containing barium sulfate filler
- A nickel plated (interior and exterior surfaces), brass, primer cup
- A brass Boxer cartridge
- A non toxic (Ti,Zn) primer tagged with gadolinium
- Double base ball powder

Re-Loaded/Re-Manufactured/Modified Ammunition

Re-loaded ammunition can include any type of commercially available primer. Hence both lead-containing and lead-free primers can be used.

In the US, lead-free reloading of hunting ammunition is growing due to increase of designated lead-free hunting grounds and regulations in some jurisdictions requiring lead-free hunting.

Tungsten bullets and other “exotic” reloading materials have also been encountered. For example:



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- .22 caliber, lead, round-nosed bullet modified with a pointed tungsten carbide core
- cartridge cases from .223 cal Remington (5.56x45 NATO) customized for a full metal jacket 7.62 Tokarev (7.62x25) bullet, unusual primers incorporating arsenic sulfide, also known as “realgar” and potassium chlorate and an anvil consisting of a steel wire, assembled into the primer cap, covered with a thin aluminium foil

Suggested Reading

Brozek-Mucha Z, Jankowicz A. Evaluation of the possibility of differentiation between various types of ammunition by means of GSR examination with SEM-EDX method. *Forensic Sci International* 2001; 123: 39.⁹³

Coumbaros J, Kirkbride KP, Kobus H and Sarvas I. Distribution of lead and barium in gunshot residue particles derived from 0.22 caliber rimfire ammunition. *J Forensic Sci* 2001; 46(6): 1352-1357.⁹⁴

Lebiedzki J, Johnson DL, Handguns and ammunitions indicators extracted from the GSR analysis. *J Forensic Sci* 2002; 47: 483.⁹⁵

Meng H-H, Caddy B. Gunshot residue detection: A review. *J Forensic Sci* 1997; 42: 553.⁹⁶

Meng HH and Lee HC. Elemental Analysis of Primer Mixtures and Gunshot Residue from Handgun Cartridges Commonly Encountered in Taiwan. *Forensic Sci J* 2007; 6(1): 39-54.⁹⁷

Wallace, J. S., Chemical Aspects of Firearms Ammunition, *AFTE Journal*, 1990 Volume 22, Number 4 (Fall), Page 364 thru 389.⁹⁸



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APPENDIX C TESTIMONY

Objectives and Goals

Forensic science is the application of a broad spectrum of sciences to answer evidential questions of interest for the legal system. Typically these questions relate to the examination and comparison of biological, trace, impression, firearms, and other physical evidence recovered in criminal investigations. GSR analysis is usually considered to be trace evidence and is analyzed with instrumentation and procedures typically found in trace evidence laboratories. Expert testimony is provided to the criminal courts by examiners that have the knowledge, skills, education, training, and experience to aid the judge or jury in understanding the science utilized in a case and the analytical results.

GSR testing is done at private, local, state, and federal laboratories. Testimony has been accepted in state and federal courts based on current methods of examinations since the 1970s.

Professional standards and practices have been proffered through national and international symposia. This guideline is intended to ensure quality and consistency in the dissemination of forensic GSR information through testimony. It is intended to assist the GSR examiner in preparing and providing effective testimony to the criminal justice system.

Ethics and Professional Obligations

Every gunshot residue analyst has an ethical responsibility to preserve the interest of science and justice. This responsibility encompasses due care taken in making scientific examinations, reports, conclusions, and interpretations. The examiner must conduct an analysis in a scientific manner, record and document findings, as well as evaluate and interpret conclusions to a submitting agency and a court of law. The interpretation and evaluation of results may be described in a written document or reported orally in a judicial proceeding. The examiner must be true to his oath and provide only those opinions that have a scientific basis. He must be guided by his knowledge, training, experience, and experimental based scientific literature in the field when interpreting findings. Interpretations of data and expert opinions should be derived from in-depth knowledge of the subject matter.

Practices that should be avoided:

- assigning greater significance to a finding or interpretation than is justified by scientifically derived data



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- the use of terminology that could be misinterpreted by a lay juror and therefore can add undue weight to the significance of a finding
- answering questions in a biased manner in any judicial proceeding
- providing testimony that is intended to mislead the court either by willful omission or by obscuring relevant information
- providing testimony outside one's area of expertise
- embellishment of one's qualifications

The analyst should follow the accepted practices of the profession and ethical codes that are delineated by their professional affiliations.

Preparation to testify

A personal Curriculum Vitae (CV) or Statement of Qualifications should be prepared before the examiner testifies. A standardized document may be required by one's individual laboratory. A Statement of Qualification form is available from ASCLD LAB on their website at www.ascl-d-lab.org. This document should include sufficient statements of education and experiential credentials to satisfy the court that the examiner is qualified to provide opinion evidence as an expert witness in gunshot residue analysis. The examiner must be prepared to explain, detail, and defend any statements in his/her CV under both direct and cross examination.

The examiner in gunshot residue should be prepared to discuss

- results and conclusion of report
- the history of forensic gunshot residue testing
- the theory of the formulation and dispersal of gunshot residue
- the instrumentation and the theory behind its use in gunshot residue analysis
- quality assurance practices of the laboratory relating to evidence handling, continuity, and validation of the technique and instrumentation used for analysis
- accreditation and certification requirements, if applicable
- the examiner's laboratory procedures.

The examiner must remain current in his knowledge of the literature and his understanding of gunshot residue analysis and interpretation.

The following is a list of documents that may be helpful to the examiner while testifying



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- a copy of report, notes, and data related to the case
- a copy of laboratory accreditation licenses or certifications
- a copy of the examiner's curriculum vitae
- a copy of relevant papers and documents that might be used by prosecutors or defense attorneys in the court case
- visual aids or displays to assist the jury in understanding gunshot residue
- a glossary of terminology used in testimony for the court reporter

Any item or document that is used as part of the testimony in court could be admitted into evidence and might not be returned to the witness.

The examiner should be prepared to address hypothetical situations based on case scenarios that might affect the interpretation of gunshot residue findings. Frequently posed hypothetical questions involve issues of contamination, persistence, misidentification, transference of gunshot residue, and whether or not the analytical findings tend to support or refute a particular scenario.

The examiner should be prepared to address controversial media articles concerning gunshot residue analysis and responses to these articles. It may be helpful to be familiar with court admissibility challenges from other jurisdictions and challenges relating to related disciplines of forensic evidence.

The examiner should try to contact the attorney or attorney's representative who is requesting testimony to determine a realistic date, time, and length of testimony.

A pretrial conference with attorneys to discuss the relevant facts of the case is important to the interpretation of results within the context of that case. The strengths and limitations of the interpretation of the gunshot residue results should be emphasized. This is an opportunity to educate the attorney prosecuting or defending an individual.

General guide to courtroom testimony and etiquette

Know the regional, state, federal rules regarding communication about the case. This can affect the ability to

- talk to others involved in the case
- observe testimony in the courtroom during the case
- talk to media or other members of the public.



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- know the responsibility and legal rights of an expert witness

Use professional demeanor and etiquette by

- dressing appropriately
- being careful in the use of humor
- speaking with respect to attorneys, judge, victim, jury, and defendant

Nothing should distract the juror's attention away from the testimony of the expert witness.

Expert witnesses act as educators to a jury. The expert has the right to explain an answer where the answer might be misinterpreted.

- Articulate the foundation of the science in a manner that a jury can understand.
- Do not talk down or belittle a jury.
- Establish eye contact with jury when explaining findings.
- Be complete in your explanations within the context of the question.
- Understand the question being asked.
- If the questioned is not understood, ask for clarification.
- It is acceptable to say "I don't know" to a question if you do not know the answer.
- Do not testify beyond your expertise.

When stating qualifications before a jury, the expert witness should follow these guidelines:

- Be accurate and thorough, emphasizing qualifications in gunshot residue.
- Expect to defend your qualifications (voir dire).
- State accreditation, if applicable, (ASCLD-LAB International, FQSI, A2LA, or ISO).

Chain of custody establishes the integrity of the evidence from the time of collection to the time of testimony. The analyst can be requested to testify to the chain of custody while in the possession of the laboratory.

Visual aids can assist the expert in educating the jury. Visual aids should represent as close as possible the actual physical evidence in a case. Analogies can also be helpful in explaining the distribution and transfer of gunshot residue.

The purpose of expert testimony is to relate the results and conclusions of the analysis to the judiciary.



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The weight of the findings is usually determined by the facts of the case. The expert may be questioned about other hypothetical scenarios and asked about the weight of the evidence in these circumstances.

Opposing expert testimony or contradictory expert opinion may be presented in a case. When it is anticipated that the opposing testimony is by an individual who is not qualified or the information given by this individual is not generally accepted in the field, then, consider

- monitoring the testimony given in the case.
 - Have a qualified analyst listen to the testimony from the audience.
 - Act as advisor to the attorney.
- researching the qualifications and obtain prior testimony given by this expert.

Some opposing attorney experts give valid testimony. In some cases, experts will not agree with each other. The weight of the testimony will be decided by the jury or trier of fact.

The expert witness should resist overstating or understating the conclusions of an examination. Facts that are combined in a manner that purposefully misleads the jury is unethical.

Preparing for an Admissibility Hearing in Gunshot Residue Analysis

Different judicial jurisdictions can have variations in the admissibility of evidence. It is important for the analyst to understand the local judicial rules for admissibility. Also, a motion to exclude evidence based on Frye or Daubert can be focused on one or more issues in the case. Responses by the forensic scientist should focus on these particular issues. Whenever possible, the expert witness should obtain a copy of the request to exclude expert testimony from the attorney. A summary of different legal rulings and requirements for admissibility are described in the section titled *Case Law on Admissibility of Expert Testimony*.

The forensic examiner must be able to assist the proponent of the physical evidence by being his conduit into the scientific community. The examiner may be asked to do this through testimony in a formal court hearing, a deposition, or by writing a declaration in support of the scientific methodology and/or conclusions.

A formal statement that the technology is not new or novel may also be appropriate.

There are a number of criteria that can be addressed in an admissibility hearing, including the following:

- General acceptance



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- peer reviewed
- relevant scientific community
- Reliability
 - can be scientifically reproduced
 - standards and controls
- Error rates
- Provides useful information (probative value is not overborne by its prejudicial effect)
- Based on sufficient facts or data
- Used the correct procedures in this case
- Expert is qualified to give an opinion on the subject

Statement regarding general acceptance in relevant community

Resources that can be used in making statements of general acceptance can include the following:

- Gunshot residue analysis by SEM/EDS has been done in forensic laboratories since the late 1970s. The foundational research report was initially published in 1977 by G. M. Wolten et al. of the Aerospace Corporation.⁹⁹ Subsequently, segments of this report were published in the Journal of Forensic Science.^{100 101 102}
- In 1994, a standard method for the analysis of gunshot residue by scanning electron microscopy/energy dispersive spectrometry was published by the ASTM as Standard E1588.
- In 2005, the Federal Bureau of Investigation hosted a symposium on gunshot residue in Quantico Virginia. Invitations were sent to private, local, state, national, and international laboratories that performed SEM/EDS analysis for gunshot residue. At the meeting, the group unanimously agreed that this technique can identify gunshot residue particles containing lead, barium and antimony. The proceeding of this meeting was published in the FBI Communications in 2006.¹⁰³
- In June of 2006, the FBI laboratory discontinued GSR analyses. The following personal communication to answer questions and concerns about this decision by the FBI was sent to the Forensic-SEM listserve. The following is the complete text of this message.

▪
From: "LeBeau, Marc A." ...



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Sent: Jun 6, 2006 12:41 PM
To: DWard ...
Subject: Message for Forensic-SEM listserv

In the May 26th issue of the Baltimore Sun, an article entitled "FBI lab scraps gunfire residue" by Julie Bykowitz was printed. I would like to take this opportunity to correct some misleading impressions that this article may have made.

While it is true that the FBI Laboratory has discontinued its gunshot residue (GSR) program, this decision was made solely on the limited number of requests for this examination and was in no way related to the GSR symposium that we hosted in 2005 or testing of our facility for the presence of gunshot residue. For the past 4 years, the FBI Laboratory has received less than ten cases per year from our field offices and other federal agencies. Consequently, the decision was made to direct our resources to areas that are related to the FBI's number one priority of combating terrorism.

In 2005, the FBI Laboratory hosted a symposium on GSR. Participants from state, local, private, and other federal laboratories participated in the symposium to identify consensus in certain areas of the GSR examination and reporting processes. We are publishing the results of that symposium in the next issue of Forensic Science Communications. However, there were no significant observations or discussions at that symposium that played any role in the decision to discontinue this service in the FBI Laboratory.

In the summer of 2005, as part of a limited training exercise for a new employee, the areas of two different units in the FBI Laboratory building were sampled for the presence of GSR. One of the units involved in this exercise was the Firearms and Toolmarks Unit where weapons are test fired and analyzed. It is important to note that this is not the unit that conducts the GSR examinations in our laboratory. As part of this training exercise, the laboratory and office spaces of the personnel assigned to the Firearms and Toolmarks Unit were sampled and tested. Because of their intimate contact with firearms, this unit was expected to contain high levels of GSR and the trainee's analysis confirmed this suspicion. However, sampling of items for GSR, as well as GSR analyses, were traditionally conducted in the Chemistry Unit in an area that was about 200 yards - or the length of two football fields - away from the Firearms and Toolmarks Unit. This unit was also sampled and tested and the Chemistry Unit's examination areas proved to be negative for GSR. Further, contamination was checked with every case that was analyzed for GSR through the use of "negative control" samples carried through the examination process. These control samples proved that any GSR detected on evidentiary items did not come from the analytical process.

Finally, I would like to emphasize to that the FBI Laboratory stands behind all of the GSR reports that it has ever issued. If requested, our experts will testify to the reports that have already been issued. Should a future FBI case require GSR analysis, the FBI Laboratory will provide a list of other federal, state, local, and private laboratories that continue to offer the GSR examination.

Further, we consider this a valuable examination that can provide valuable probative evidence in criminal investigations.

Sincerely,
Marc A. LeBeau, Ph.D.



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Chemistry Unit Chief FBI Laboratory

- A scientific working group on gunshot residue (SWGGSR) was formed in 2007 to
 - Promote professional development in gunshot residue analysis
 - Provide a means of information exchange with the international forensic science community
 - Provide guidelines for gunshot residue investigations, examinations, and reporting
- Other published bibliographies.

Statement of reliability

Supporting material or documents for the reliability of gunshot residue may include the following:

- Validation studies, Proficiency Tests, Competency Tests
- Accredited discipline within ASCLD/ LAB International
- Existence of published standardized methodologies – ASTM, SWGGSR, ENFSI
- Reliability of instrumentation as validated and certified by the manufacturer
- Describe controls and standards used in the analysis
- Existence of searchable libraries, if applicable
- Literature concerning reliability of gunshot residue testing
- Knowledge of utilization of this technology by other experts, both within the field of forensic science and in other areas of science
- Ability to reanalyze a sample by an independent laboratory

Error Rate Statement

The determination of error rates as one of the delineated Daubert guidelines for determining admissibility is ambiguous. Error rates determination may or may not be an issue in any specific motion. Refer to the language in the motion to address the specific error rate to be discussed.

Provides useful information where its probative value is not overborne by its prejudicial effect.



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The examiner is responsible for communicating to the court not only the finding in a case but its significance or lack thereof.

Based on sufficient facts or data

The examiner must be able to define or cite observations, procedures, methods, data, scientific studies, and in some instances case-derived information or facts that formed the basis of his conclusions or interpretations.

Used the correct procedures in this case

The examiner should be able to communicate that he used established, validated laboratory procedures in the case. Discuss the review policies of the laboratory including procedures for a

- technical review by a qualified peer
- administrative review for compliance to laboratory policy

If applicable, a statement concerning awareness of any errors, breaches of protocols, sample contamination, or other extraneous factors that would question the validity of the conclusions may be appropriate.

Expert witness is qualified to give an opinion on the subject

In an admissibility hearing, a complete and thorough description of one's qualifications (voir dire) and expertise is essential. This description is often more extensive than is used to testify in a general court proceeding. Consider describing in detail the following:

- general qualifications – education, training, and experience in the analysis and interpretation of the evidence being challenged
- background information of the forensic laboratory, including staffing, areas of expertise, area serviced, longevity
- accreditation and certification programs
- professional development of the expert – memberships, subscription to relevant journals in the field of interest, participation in seminars and workgroups (SWG and TWG groups)
- publications, lectures, and presentations
- training, teaching, and research in the area of interest
- statement of familiarity with current standards and practices in the area of interest.



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Case law on admissibility of expert testimony

The rules of evidence governing the admissibility of expert testimony will differ by jurisdiction, and GSR examiners are advised to consult the rules of evidence and pertinent case law applicable to the court in which they will be giving evidence. In the United States, federal courts follow the Federal Rules of Evidence, while state courts generally follow rules of evidence imposed by state law. Not all states currently have the same rules for challenging the validity of the science underlying the expert opinion of the witness nor do all impose the role of gatekeeper upon the judiciary. In responding to challenges regarding admissibility, the witness should prepare his response specific to the jurisdiction where the testimony is to be given and address the specific issue or issues raised in the challenge.

The rules of admissibility for Expert Testimony in Federal court were amended in 2000 in response to **Daubert v. Merrell Dow Pharmaceuticals, Inc., 509 U.S. 579 (1993)**, and to the many cases applying Daubert, including **Kumho Tire Co. v. Carmichael, 119 S.Ct. 1167 (1999)**.

Daubert v. Merrell Dow Pharmaceuticals, Inc. 509 U.S. 579 (1993) gives the court the responsibility of evaluating expert testimony for compliance to the Rule 702. Over the years, subsequent court decisions, such as **Kumho Tire Co., Ltd. V. Carmichael, 526 U.S. 137 (1999)** and **General Electric Co. v. Joiner. 522 U.S. 136 (1997)**, have modified Daubert. The basic concepts in Daubert that the forensic scientist today needs to address are listed below.

- Is the reasoning or methodology scientifically valid and reliable?
- Daubert defines five non-binding factors to consider in admitting evidence. These are intended as a guide for the court to consider in making an admissibility decision.
 - Can theory or technique be tested scientifically?
 - Has process been subjected to peer review?
 - What is the known or potential error rate of the technique or theory?
 - Are there standards controlling the technique's operation and maintenance?
 - Is it accepted by the relevant scientific community?



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Additional admissibility criteria arising from other federal decisions (reference Commentary to the 2003 version of the Federal Rules of Evidence regarding Rule 702):

- Whether expert testimony is a product of research the expert witness conducted independent of the litigation, or whether he developed his opinion expressly for purposes of testifying. (Daubert)
- Whether the expert has unjustifiably formed an unfounded conclusion from an accepted premise. (See **General Elec. Co. v. Joiner**, 522 U.S. 136, 146 (1997).
- Whether the expert has adequately accounted for obvious alternative explanations. (See **Claar v. Burlington N.R.R.**, 29 F.3d 499 (9th Cir. 1994).
- Whether a field of expertise is capable of reaching reliable results and whether the opinion given by an expert is consistent with type of opinions that experts in this field would typically give. (See **Kumho Tire**, 119 S.Ct.at 1175; see also **Moore v. Ashland Chemical, Inc.**, 151 F.3d 269 (5th Cir. 1998).

According to **Federal Rule of Evidence 702**, admissibility of evidence or a fact in issue is determined by “expert testimony given to assist trier of fact to understand evidence.” Three prongs are defined in **Rule 702** as its relevance and reliability standard.

- Testimony is based on sufficient facts or data.
- Testimony is a product of reliable principles and methods.
- Expert must apply reliable principles and methods.

The following are Excerpts from the Federal Rules of Evidence as amended to December 1, 2008:

- **Rule 702.** Testimony by Experts

“If scientific, technical, or other specialized knowledge will assist the trier of fact to understand the evidence or to determine a fact in issue, a witness qualified as an expert by knowledge, skill, experience, training, or education, may testify thereto in the form of an opinion or otherwise, if (1) the testimony is based upon sufficient facts or data, (2) the testimony is the product of reliable principles and methods, and (3) the witness has applied the principles and methods reliably to the facts of the case.

(As amended Apr. 17, 2000, eff. Dec. 1, 2000.)”

- **Rule 703.** Bases of Opinion Testimony by Experts



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“The facts or data in the particular case upon which an expert bases an opinion or inference may be those perceived by or made known to the expert at or before the hearing. If of a type reasonably relied upon by experts in the particular field in forming opinions or inferences upon the subject, the facts or data need not be admissible in evidence in order for the opinion or inference to be admitted. Facts or data that are otherwise inadmissible shall not be disclosed to the jury by the proponent of the opinion or inference unless the court determines that their probative value in assisting the jury to evaluate the expert’s opinion substantially outweighs their prejudicial effect.

(As amended Mar. 2, 1987, eff. Oct. 1, 1987; Apr. 17, 2000, eff. Dec. 1, 2000.)”

The following sections include admissibility rules for some states that do not follow the Federal Rules of Evidence.

- **Frye States** - Frye states follow the ruling set forth in **Frye v. United States (293 F. 1013 (DC Cir 1923)) District of Columbia Circuit Court in 1923**). This legal decision says that when novel scientific evidence is an issue, Frye allows the judiciary to defer to scientific expertise to determine if it has gained general acceptance in the relevant field. The case pertained to the admissibility of polygraph evidence in court. “Just when a scientific principle or discovery crosses the line between the experimental and demonstrable stages is difficult to define. Somewhere in this twilight zone, the evidential force of the principle must be recognized, and while the courts will go a long way in admitting experimental testimony deduced from a well-recognized scientific principle or discovery, the thing from which the deduction is made must be sufficiently established to have gained general acceptance in the particular field in which it belongs.” Essentially the Frye Ruling must meet general acceptability in the scientific community, thereby excluding novel techniques. i.e. polygraph examination. In most but not all jurisdictions, the Frye standard has been superseded by the Daubert standard.
- **Frye / Mack States** -: Minnesota's Dominant Standard has long recognized the Frye "general acceptance" test as the proper standard for the admissibility of scientific expert testimony in the state's courts. Specifically, in **State v. Mack (292 N.W.2d 764, 768 Minn. 1980)**, the Minnesota Supreme Court formulated the current approach to the Frye inquiry as follows:



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- Under the Frye rule, the results of mechanical or scientific testing are not admissible unless the testing has developed or improved to the point where experts in the field widely share the view that the results are scientifically reliable as accurate.
- The Minnesota Supreme Court in **State v. Schwartz (447 N.W.2d 422, 424 Minn. 1989)** indicated that, “we have rephrased the Frye standard to require that experts in the field generally agree that the evidence is reliable and trustworthy.”
- **Kelly/ Frye States** - The Kelly standard, in California, as described in **People v. Kelly (Cal. 1972), 549** modifies Frye in that evidence based upon application of a new scientific technique may be admitted
 - after the reliability of the scientific technique has been foundationally established, usually by the testimony of an qualified expert witness.
 - the scientific technique being offered must have gained general acceptance in the particular field to which it belongs.
 - the court merely determines if there is a general consensus by surveying a cross-section of the relevant scientific community, literature, and the expertise of public opponents of the technique.
- **Tennessee** has rejected Frye and follows a test similar to Daubert. [McDaniel v. CSX Transp., 955 S.W.2d 257 \(Tenn. 1997\), cert. denied, 524 U.S. 915 \(1998\).](#) In **McDaniel v. GSX Transportation, Inc., 955 S.W. 2d 257 (1997)**, the Tennessee Supreme Court listed five nonexclusive factors taken from the federal case of **Daubert v. Merrell Dow Pharmaceuticals, 509 U.S. 579 (1993)**:
 - “(1) whether scientific evidence has been tested and the methodology with which it has been tested;
 - “(2) whether the evidence has been subjected to peer review or publication;
 - “(3) whether a potential rate of error is known;
 - “(4) whether, as formerly required by Frye, the evidence is generally accepted in the scientific community; and
 - “(5) whether the expert’s research in the field has been conducted independent of litigation.”

★ **Canadian Case Law On Admissibility Of Expert Evidence:**

- **R. v. Mohan (1995), 37 C.R. (4th) 395 (Ont. C.A.)**



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Admission of expert evidence depends on the application of the following criteria:

- Relevance - The evidence must be logically and legally relevant. Its probative value must outweigh its prejudicial effect. Its delivery should not involve an inordinate amount of time in relation to its value. Its reliability must exceed its potential to mislead.
- Necessity in Assisting the Trier of Fact - The evidence is likely to be outside the experience and knowledge of a judge or jury. It must be necessary to enable the trier of fact to appreciate the matters in issue due to their technical nature. The subject matter must be such that lay people would be unlikely to form a correct judgement about it unless assisted by persons with special knowledge.
- The Absence of any Exclusionary Rule - To ensure the admissibility of expert evidence, it cannot run afoul of an exclusionary rule of evidence separate and apart from the opinion rule itself.
- A Properly Qualified Expert - The evidence must be given by a witness who is shown to have acquired special or particular knowledge through study or experience in the matters on which he or she undertakes to testify.

- **R. v. B. (L.) 35 O.R. (3d) 35 (Ont.C.A.),**

When considering the probative value of proposed evidence, consideration should be given to such matters as

- the strength of the evidence
- the extent to which the proposed evidence supports the inference(s) sought to be made from it (this factor will often correspond to the degree of similarity between the prior misconduct and the conduct forming the subject-matter of the charge)
- and the extent to which the matters it tends to prove are at issue in the proceedings.

★ **The following are excerpts from some state evidence codes regarding testimony by expert witnesses:**

- **California Evidence Code** defines the following rules of evidence:

801. “If a witness is testifying as an expert, his testimony in the form of an opinion is limited to such an opinion as is:

- (a) Related to a subject that is sufficiently beyond common experience that the opinion of an expert would assist the trier of fact; and



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(b) Based on matter (including his special knowledge, skill, experience, training, and education) perceived by or personally known to the witness or made known to him at or before the hearing, whether or not admissible, that is of a type that reasonably may be relied upon by an expert in forming an opinion upon the subject to which his testimony relates, unless an expert is precluded by law from using such matter as a basis for his opinion.”

802. “A witness testifying in the form of an opinion may state on direct examination the reasons for his opinion and the matter (including, in the case of an expert, his special knowledge, skill, experience, training, and education) upon which it is based, unless he is precluded by law from using such reasons or matter as a basis for his opinion. The court in its discretion may require that a witness before testifying in the form of an opinion be first examined concerning the matter upon which his opinion is based.”

803. “The court may, and upon objection shall, exclude testimony in the form of an opinion that is based in whole or in significant part on matter that is not a proper basis for such an opinion. In such case, the witness may, if there remains a proper basis for his opinion, then state his opinion after excluding from consideration the matter determined to be improper.”

- **Indiana Rules of Evidence**

Rule 702. Testimony by Experts

(a) If scientific, technical, or other specialized knowledge will assist the trier of fact to understand the evidence or to determine a fact in issue, a witness qualified as an expert by knowledge, skill, experience, training, or education, may testify thereto in the form of an opinion or otherwise.

(b) Expert scientific testimony is admissible only if the court is satisfied that the scientific principles upon which the expert testimony rests are reliable.

Rule 703. Bases of Opinion Testimony by Experts

The facts or data in the particular case upon which an expert bases an opinion or inference may be those perceived by or made known to the expert at or before the hearing. Experts may testify to opinions based on inadmissible evidence, provided that it is of the type reasonably relied upon by experts in the field.



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Typical questions and example answers used in examination of an expert witness in gunshot residue

Note: These are only sample questions and answers that can be given in the course of court testimony. Individual responses will vary depending on each laboratory's procedures and policies. GSR in these responses relate to primer gunshot residue analyzed by scanning electron microscopy/energy dispersive spectrometry (SEM/EDS).

- ★ **What is GSR?** Gunshot residue (GSR) is the chemical compounds and particulate materials that result when a firearm is discharged. Most of the residue that comes out of the barrel of the gun is burned, unburned, and partial burned propellant (organic) and bullet fragments. This material is most often used to determine the distance between the muzzle of a gun and a target. In addition, some gunshot residue is produced from the detonation of the primer mixture during the discharge. This residue is typically collected on adhesive lifts from the hands of a suspected shooter or other objects. The typical characteristics of gunshot primer residue are
- **Size** – These condensed primer residue particles are typically in the range of 1 to 10 micrometers in size. In comparison, an average human hair is 100 micrometers in diameter.
 - **Chemistry** – Most primers used in North America consist of lead styphnate as an initiating explosive, barium nitrate as an oxidizer, and antimony sulfide as a fuel. Therefore we commonly look for lead, barium, and antimony, or combinations thereof, in a single particle. Other primer mixtures without these elements also exist.
 - **Morphology** – These particles are typically spheroid or show shape characteristics of having been molten.
- ★ **Explain what instrumentation is used in GSR analysis?** Scanning electron microscopy / energy dispersive spectrometry (SEM/EDS) is used to analyze adhesive lifts from suspected shooters. A high powered electron microscope (SEM) is used to find very small particles. An energy dispersive spectrometer (EDS) is used to identify a particle's elemental composition. The spectrometer detects x-ray energy generated by the interaction of the electron beam with the particle. This method (SEM/EDS) has been around for a long time and has been utilized in GSR analysis since the 1970s.



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- ★ **Are scanning electron microscopes widely accepted in the scientific community?**
Absolutely! The scanning electron microscope, or SEM, has enabled scientists of numerous disciplines to observe and analyze very small things with tremendous resolution. It has been an analytical tool in science since the early 1960s. The SEM/EDS is used to identify explosives, paint, GSR, and many other particles in forensic cases. It has been used in GSR analysis since the 1970s. In 2005, over 200 SEMs were being used in forensic laboratories and over 130 of them were being used for GSR analysis. Today, the numbers continue to grow.

- ★ **What is the theory behind SEM/EDS?** – A high energy electron beam is produced by heating a filament (like an incandescent light bulb) and forcing the electrons down a column while focusing them to a very small diameter beam. Many interactions occur as the high energy electron beam penetrates into the sample surface. Electrons from the atoms comprising the sample are ejected from their orbits around the atomic nucleus and detected. As an analogy, picture the Earth (as an electron) being ejected from its orbit around the sun (as the nucleus). The ejected electrons generate an extremely high resolution image on the SEM computer monitor capable of tremendous magnification, and a set of x-rays whose energies vary according to the chemical elements present. The detected x-rays come from the energy released when an outer shell (orbit) electron replaces an electron knocked out of its inner shell by the focused beam. The EDS computer monitor shows a spectrum that is a plot of x-ray energies (peaks) on one line (x-axis) and the total counts of each x-ray energy on the second perpendicular line (y-axis). The operator can then identify the elemental make-up from its characteristic spectrum. In summary, a high powered microscope enables us both to see very small particles like GSR and then to identify the elements in the particle like lead (Pb), barium (Ba), and antimony (Sb).

- ★ **Explain what software is used in GSR analysis?** Several companies make energy dispersive x-ray spectrometers. Each company has its own software package to help in the identification of inorganic elements and compounds found with the scanning electron microscope. Some have software packages specifically designed to analyze GSR.

- ★ **How is GSR analysis automated?** Automated software packages can take control of the stage on the SEM and automatically examine a sample for a pre-determined set of elements like Pb, Ba, Sb. The analyst can set search parameters such as magnification, speed, termination limits, and others to allow for optimum identification of GSR.

- ★ **What control samples are used in GSR analysis?** A known reference sample with lead, barium, and antimony or a material similar in atomic number is examined with



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each automated analysis for quality assurance purposes. The known reference sample is analyzed before and after the case samples in the automated analysis. A reproducible number of GSR particles in the size range of interest should be found to ensure the instrument is working properly from beginning to end. The second type of reference sample is a cobalt and rhodium standard used to set the brightness and contrast to the same level for each analysis. In addition, a cobalt or copper or similar type reference sample is analyzed before each batch of samples to check that the instrument is properly calibrated and properly identifying elements.

- ★ **What Standard Operating Procedures are used in GSR analysis?** My laboratory has a written standard operating procedure for gunshot residue analysis. The procedure includes sample preparation and identification, setup of the SEM parameters, setup of the automated analysis, and confirmation of the GSR particles, printing, storing and reporting of data.
- ★ **What assurances do you have that there is no laboratory contamination?** In order to ensure no contamination of GSR case samples, my laboratory has certain criteria in place. Samples to be tested for GSR are never exposed to the firearms area of the laboratory. Sample stubs are only exposed to the air immediately before and after being placed in the SEM vacuum chamber. I have a known blank adhesive lifter that was exposed to the same air at the same time as my samples and was found to be negative for GSR particles. I monitor the examination area by placing a blank adhesive lifter in the laboratory where I test clothing for GSR. This is done before examining clothing in this area. Clothing samples, or fibrous samples, are analyzed alone without other case samples in the vacuum chamber.
- ★ **What is the history of GSR analysis?** Teodoro Gonzalez from Mexico introduced the “Gonzalez Test” to the Milwaukee Police Dept. in 1933. This test later became known as the “Dermal Nitrate Test” and the “Paraffin Test.” A diphenylamine and diphenylbenzidine reagent was used to detect nitrates and nitrites on hands of suspected shooters. These tests were used in the 1960s. But, because of the occurrence of false positive and false negatives, the dermal nitrate test is considered to be unreliable in determining the presence of gunshot residue on a subject’s hands. Neutron Activation Analysis became popular in the late 1960s and early 1970s. This technique quantitatively determines the levels of antimony and barium on hand swabs. It was quickly replaced by the less expensive Atomic Absorption method of analysis. In 1977, the Aerospace Report on Particle Analysis for Gunshot Residue Detection presented a comprehensive guide for the analysis of GSR particles by SEM/EDS. This has become the preferred method of analysis for GSR in most forensic laboratories.



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- ★ **How are GSR particles produced, transferred, and deposited?** The firing pin hits the back of the cartridge activating the shock sensitive primer. The primer ignites the propellant (gunpowder) that forces the bullet down the barrel of the gun and on its path. Metals from the primer are vaporized by the heat and pressure within the cartridge. Vapors escape from any area of the weapon that are not gas tight, like the breach area and muzzle. The heat of this explosive reaction results in tiny metal containing particles. These particles fall on anything in the vicinity of the fired weapon, including the hands of the shooter.
- ★ **What can you say about finding GSR or not on a person's hands?** The presence of gunshot primer residue on a person's hand is consistent with that person
 - having discharged a firearm,
 - having been in the vicinity of a firearm when it was discharged,
 - and/or having handled an item with gunshot primer residue on it.

The witness should not give undue weight to one conclusion over another. The significance of any finding is dependent on the circumstances of any case.

Correspondingly, when no GSR is found on the hands of a suspect, one cannot preclude the possibility that a person fired a gun. There are many reasons why suspects may not have GSR on their hands even if they discharged a firearm. The most common reasons for not detecting residue on a known shooter's hands are

- washing the hands after the shooting,
 - a long delay between the shooting incident and collecting the samples,
 - the weapon or ammunition used in the shooting incident do not deposit significant amounts of detectable residue.
- ★ **What visual aids are helpful during testimony?** It could be helpful to bring to court a photograph or digital image that depicts the visible smoky or sooty material emanating from a gun that has been discharged. This will give jurors an understanding of the dispersion of GSR from a gun when it is fired.
 - ★ **Are there any helpful analogies when explaining GSR?**
 - Human hair is typically 100 micrometers in diameter and the particles we find are typically 1-5 micrometers in size.



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- If the SEM stub was the size of two football fields side by side, we would be looking for a particle the size of a pea.
- If you put flour on your hands and clap your hands in a sunlit room, the fine dust you see floating and slowly falling to the ground is like GSR falling after a fired weapon.
- ★ **How easily is GSR removed?** Since GSR is microscopic sized solid particles lying on surfaces in the vicinity of where a weapon had been fired, they can easily be removed. Normal activities people do with their hands can wipe these particles away, like putting one's hands in pockets, handling items, or rubbing hands together. Washing hands will remove most if not all particles.
- ★ **What are the post shooting time requirements for collection of GSR?** Rates of particle loss vary widely with the activity of the subject. Because particles are easily wiped off with the normal things people do with their hands, we expect particles to be gone within 4 to 5 hours after a shooting event.
- ★ **Where does lead, antimony, barium occur other than in GSR?** Combinations of lead, barium, and antimony have been reported in a few other sources. Some of these include commercial fireworks or pyrotechnics, air bags, and brake linings. Typically, residue from these materials has different morphology or additional elements not typical of gunshot residue. These additional elements allow these particles to be distinguished from gunshot residue particles.
- ★ **How do law enforcement officers or crime scene investigators collect a GSR sample?** Police officers are trained to collect samples as soon as possible and preferably before transportation to the police station. They are trained to wear gloves when sampling suspects and are trained to prevent contamination.
- ★ **How likely is law enforcement as source of contamination in GSR collection?** Tests I have done on the hands of police officers, along with the research of others in this field, show that contamination from police officers hands is unlikely. In fact, most officers when tested were found to have no particles on their hands. With the exception of a police officer who has recently fired his weapon, the few officers that do have particles on their hands are unlikely to transfer more than one or two particles to a subject that they have touched. One reason can be that they wash their hands more often than they touch their firearms.
- ★ **Can GSR particles be transferred from a shooter or another object contaminated with GSR to someone else that did not fire a gun?** Yes. The amount will depend on



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the number of GSR particles on the contaminated surface or the shooter's hands. The number of particles transferred will be a small percentage of the total number of particles present.

- ★ **What about contamination from sources outside the laboratory?** Contamination from other sources outside of the laboratory is beyond the analyst's knowledge and control. The analyst can only testify to any training given to outside agencies that address contamination.
- ★ **How is gunshot residue dispersed when a weapon is fired?** Most of the gunshot residue particles escape from the muzzle and chamber area of a firearm. Gunshot residue particles can travel several yards downrange. These particles tend to follow the path of the bullet as it travels downrange. The distance will depend on the type of weapon, caliber, and environmental conditions. In addition, long guns, like rifles and shotguns, tend to leave less GSR on shooters than handguns. Gunshot residue detected on a subject's hand by SEM/EDS should not be confused with gunshot residue used to determine the distance between the muzzle of a firearm and a target at the time of discharge. A distance determination is based on the distribution of gunpowder particles and lead and or copper residue deposited around a bullet hole. It is not possible to use GSR particles produced from the primer of a cartridge to determine a distance between the muzzle of a firearm and a target.
- ★ **How likely is a bystander to have GSR on him?** Tests show that a person standing within three feet to the side of a shooter may have GSR on their hands whereas a person standing 10 or more feet to the side of a shooter typically do not have GSR on their hands. This can vary by the type of gun and ammunition, number of shots fired, and the environment of the shooting.
- ★ **How should we answer questions about scenarios?** Listen to scenarios or hypotheticals carefully. A hypothetical or scenario can be proposed by the prosecutor or the defense with the goal of getting the witness to acknowledge the possibility that GSR results fits their proposal. Some scenarios are more reasonable than others. Answers can range from
 - yes – the results fit this hypothetical
 - yes – it is possible that the results could fit this hypothetical but it is less likely than other scenarios
 - yes – it is possible that the results fit a hypothetical but this is an unlikely situation



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- no – the results do not fit the hypothetical

It is important in responding to a hypothetical question for the witness to clearly define the parameters of the hypothetical in the answer.

- ★ **Is it possible to find gunshot residue on clothing?** Yes. Samples are collected from clothing using the same adhesive lifts as are used on the hands of subjects suspected of shooting a gun. The areas of the clothing to be sampled will depend on the likelihood of finding residue if the person wearing the clothing was firing the gun, was carrying the gun in a particular location, or was trying to conceal a gun in a particular manner. Excessively soiled or bloody areas of clothing are usually avoided since these materials can inhibit the ability to find GSR particles.
- ★ **How long will gunshot residue last on clothing?** We know from laboratory tests that GSR on clothing will last considerably longer than on hands, exactly how long is unknown. However, most if not all residue will be removed when the clothing is washed.
- ★ **How long will gunshot residue last on an object other than hands or clothing?** GSR will remain on any object until something removes it.
- ★ **Can you determine what type of weapon or ammunition was used by the type of gunshot residue detected?** I can not determine the type of weapon that was used by the elemental composition of GSR. However, the ammunition can vary in its elemental composition because of variation in the compounds used in the primer mixture. For instance, depending on manufacturer, .22 caliber ammunition can have lead and barium instead of lead, barium, and antimony in gunshot primer residue. Lead-free ammunition can have strontium or zinc and titanium present in its gunshot primer residue.



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APPENDIX D TRAINING

OBJECTIVE

To provide an outline of goals for a comprehensive training program in gunshot residue analyses by scanning electron microscopy/energy dispersive x-ray spectrometry (SEM/EDS).

SCOPE OF GUIDELINE

This guideline pertains only to the analysis of gunshot residue by manual and computer controlled SEM/EDS.

INITIAL TRAINING

- ★ Analytical methods for detection and analysis of gunshot primer residue. (Introduction)
 - Historical (such as paraffin test, NAA)
 - Current (AA, ICP, SEM/EDS)
 - Understand why SEM/EDS is the preferred method of gunshot primer residue analysis
- ★ Origin and formation of GSR. (Procedure—Samples from Ammunition and Firearms; Appendix B)
 - The role of the firearm
 - Creation of GSR
 - Deposition of GSR
 - Distribution of GSR
 - The components of the ammunition: purpose and function.
 - Bullet
 - Cartridge case
 - Propellant
 - Primer
 - 1) Initiator
 - 2) Oxidizer
 - 3) Fuel
- ★ Collection of GSR. (Appendix A)
 - The impact of collection conditions, such as:
 - Moisture
 - Blood
 - Soil
 - Weather
 - Collection media, such as:
 - Double-back carbon adhesive.



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- Freshly activated adhesive.
 - Non-conductive adhesive.
 - Common surfaces for collection.
 - Hands
 - Face
 - Other body regions, such as:
 - Nostrils.
 - Hair.
 - Arms.
 - Sampling clothes.
 - Awareness of sources of contamination
 - Sampling other inanimate objects, such as:
 - Vehicle surfaces
 - Upholstery
- ★ SEM/EDS instrument-specific operating parameters for the detection of GSR. (Instrumental Requirements)
 - Automated analysis (Procedure—Setting the Detection Parameters and Automated Analysis)
 - Relocation and manual confirmation (Procedure—Manual Examinations and Analysis of Detected Particles.
- ★ Techniques to handle charging samples. (Procedure—Sample Preparation)
 - Carbon coating
 - Variable pressure systems
- ★ Quality Assurance/ Quality Control (Calibration and Quality Assurance)
 - Laboratory Protocols
 - Appropriate blanks and environmental controls
 - Appropriate positive controls/references
- ★ Observe the analytical process performed on actual casework by a qualified GSR analyst, where available. The cases observed should involve a variety of sample types and results. Examples:
 - Varying concentrations of GSR particles on a stub (heavy to light to none)
 - “Dirty” samples (fibers, soil, etc.) to “clean” samples
- ★ Practice analyzing known positive and negative GSR samples that contain various concentrations and compositions.
- ★ Interpret results and write report. (Documentation; Reporting Criteria)



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- Know what constitutes a positive result and what it means.
 - Primary transfer
 - 1) Fired a weapon
 - 2) Was in close proximity to a firearm when discharged
 - Secondary, tertiary, etc. transfer
 - 1) Handled a weapon
 - 2) Contacted a surface containing GSR
- Know what constitutes a negative result and what it means, such as:
 - The individual/item was not associated with a firearm discharge.
 - GSR was removed before collection.
 - Weapon/ammunition does not reliably produce GSR.
- Know identification/classification of GSR particles by size/shape, morphology, and composition.
- Know how to differentiate Pb-Sb-Ba particles containing elemental tags that would indicate sources other than the discharge of a firearm.
- ★ Conduct a study related to GSR analysis, that may include projects such as:
 - Analysis of samples from brake pads.
 - Analysis of samples from fireworks.
 - Analysis of samples from persons of varying occupations.
 - Analysis of GSR samples related to time lapse between incident and collection.
 - Assessment and monitoring potential contamination issues.
- ★ Successfully complete competency testing.
- ★ Perform analyses under the supervision of a qualified GSR analyst, where available.
- ★ Testimony. (Appendix C)
 - Observe the testimony of experienced gunshot residue experts, where available.
 - Participate in a mock trial.
 - Monitor testimony.
 - Become familiar with appropriate federal and state laws governing rules of evidence and expert witness testimony
 - Become familiar with landmark decisions, e.g. Daubert and Frye

ONGOING TRAINING.

- ★ Review literature.
- ★ Attend external workshops, seminars, etc.
- ★ Attend external classes.
- ★ Participate in round-robin and/or proficiency tests.

SUGGESTED READING.



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GLOSSARY OF SEM TERMS – an extensive glossary of gunshot residue terms can be found in Swoebble and Exline’s “Current Methods in Forensic Gunshot Residue Analysis”¹²⁵

Accelerating Voltage

The voltage/electrical potential applied to the filament that accelerates the electrons emitted by the electron gun/filament. In general, increasing the accelerating voltage will decrease the spherical aberration of the system and, consequently, increase the resolution.

However, varying the acceleration voltage also affects the beam-specimen interaction. Consequently, if a higher accelerating voltage is used, the interaction volume between the beam and specimen will be bigger. This needs to be considered when analyzing small particles as the interaction volume may exceed the size of the particle.

SE images obtained using low kV accelerating voltages (5 to 10 kV) provide exceptional topographical information because the beam interaction is confined to regions very close to the surface. As a result, the yield of BSE is low using low kV accelerating voltages,

Conversely, using high kV (20 to 30 kV) accelerating voltages, much higher yields of BSE are obtained due to the deeper penetration and interaction volume of the beam.

As the accelerating voltage increases, so does the detectability of higher atomic number elements. The accelerating voltage needs to be a minimum of 20 kV to permit generation of (and detection there of) the Lead ‘L’ lines.

Atomic Number

The number of protons found in the nucleus of an atom. It is conventionally represented by the symbol **Z**. The atomic number uniquely identifies a chemical element.

Background X-rays

Also known as *Bremsstrahlung*, *braking radiation* or *continuous spectrum*. Non-specific x-ray radiation with a continuous energy range from zero up to the beam voltage. Background radiation results from the deceleration of beam



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electrons in the atomic Coulombic field. A typical x-ray spectrum will consist of both a continuous background and peaks from characteristic x-rays.

Backscattered Electrons

Abbreviated to *BSE*: High energy electrons from the primary electron beam of the *SEM* that are elastically reflected by the specimen. The probability of backscattering is proportional to atomic number: that is, elements having high atomic number are more efficient at backscattering electrons than elements of low atomic number. Therefore, the intensity of the image relates to the mean atomic weight of the sample.

Beam Current

The number of electrons available to interact with the sample.

Bulk Analysis

A term usually used to describe analysis in which all, or a large portion, of the specimen is analyzed. In *SEM*, the average elemental composition of a material is determined through the analysis of as large an area as possible of the specimen and may be achieved by a single large area raster or the summed results from multiple smaller area rasters.

The term is also used to describe the collection, pooling, and concentration of a sample from a surface such as in the sample collection for GSR by atomic absorption analysis (AAS).

Cathodoluminescence

Emission of photons in the ultraviolet, visible, and infrared regions of the electromagnetic spectrum as a result of electron beam interaction with certain materials.

Characteristic X-rays

X-ray emission resulting from de-excitation of an atom following inner shell ionization. The energy of the x-ray emission is dependent on the quantum transition within the inner electron shell. As the quantum levels are different for each element, the energies of the x-rays emitted are characteristic of a particular element. This forms the basis of x-ray analysis. A typical x-ray spectrum will consist of both a continuous background and peaks from characteristic x-rays.



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Charging	Deflection or sudden electrical discharge of the sample, resulting from negative charge accumulation on either a non-conductive sample or sample that is not properly grounded. This effect can interfere with image formation and x-ray analysis. It can usually be eliminated by application of a conductive coating.
Condenser Lens Current	<p>The current in the condenser lens changes the spot size or diameter of the electron beam. An increased condenser lens current produces a smaller spot size and, in general, results in better resolution.</p> <p>However, an increase in the condenser lens current results in a lower beam current and, therefore, fewer electrons interact with the sample. Consequently, fewer secondary and back scattered electrons become available which results in poor image quality.</p>
Energy Dispersive X-ray Spectroscopy	<p>Abbreviated as <i>EDS</i>, <i>EDXA</i> or <i>EDX</i>. X-ray Spectroscopy based upon the simultaneous measurement of the energies of x-rays emitted by a sample, generally between 0 and 20 keV.</p> <p>WDS is not used for particle detection as its response is very slow compared to EDS; however, it can be used to aid in the confirmation of the elemental composition of particles as it has superior sensitivity and resolution than EDS.</p>
Final Aperture	The last beam-restricting orifice in an electron optical column. The orifice diameter influences the beam current and depth of focus.
Gunshot residue	Abbreviated to <i>GSR</i> (also referred to as Cartridge-case Discharge Residue – CDR, or Firearm Discharge Residue – FDR). Residues formed during the discharge of a firearm. In the context of this method, GSR is the inorganic and metallic residues largely originating from the ammunition that has been discharged but may include contributions from the firearm and previous ammunitions discharged from the firearm.
Interaction volume	The sample volume penetration depth in which the electron



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beam is scattered until it loses most of its energy. It is generally thought of as the volume in which detectable x-rays are produced. The actual volume will vary depending upon beam voltage, average atomic number, and density of the sample.

The interaction volume increases with the accelerating voltage of the beam. This needs to be considered when analyzing small particles as the interaction volume can exceed the size of the particle.

Live Time

The time that the EDS electronics are available to accept and process incoming x-rays. Live time is expressed as a percentage of real time. Dead time is the opposite complement to this, i.e., 55% live time equals 45% dead time.

Major element (EDS detection)

Major elements, as determined from the x-ray spectrum of a sample, are those whose most intense x-ray peak is greater than approximately 30% of the highest peak (relative peak height).

Minor element (EDS detection)

Minor elements, as determined from the x-ray spectrum of a sample, are those whose most intense x-ray peak is less than approximately 30% but higher than approximately 10% of the highest peak (relative peak height).

Pulse Processor Time Constant

Operator selected value for pulse processing time. A higher value will result in more accurate determination of pulse height (better spectral resolution). A lower value will result in a higher count rate, but with reduced spectral resolution.

Raster

The rectangular pattern scanned by the electron beam on a sample. The raster dimensions will change inversely with magnification. The raster pattern is built up from a series of horizontal scan lines in the same way an analogue television picture is generated.



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Sample: Known	The sample obtained from the object suspected to be the source of the recovered materials. In the context of this Guide, as an example, a sample of GSR from a fired cartridge case recovered at the scene. Often referred to as <i>control sample</i> (this alternative is discouraged).
Sample: Recovered	The sample recovered from the item(s) under examination; the sample to be compared with the known or reference sample. Often referred to as <i>unknown or questioned sample</i> (this alternative is acceptable).
Sample: Reference	A sample of a particular source/origin, composition. In the context of this Guide, a sample of GSR from test firings, etc.
Scanning Electron Microscopy	Abbreviated to <i>SEM</i> . A form of microscopy employing electrons rather than visible light to obtain a high resolution image over a dynamic range of magnification.
Secondary electrons	Abbreviated as <i>SE</i> : low energy electrons emitted as a result of interaction of the primary beam electrons with conduction band electrons of atoms in the interaction volume. They are produced throughout the interaction volume but only those at or near the surface have enough energy to escape and thereby form an image.
Specimen stub	A sample holder / collection device for examination using <i>SEM/EDS/WDS</i> usually made of aluminum. The stub consists of a wide flat sample surface coated with adhesive and a pin or other type of fitting for mounting on the <i>SEM</i> stage.
Spectral Artefacts	Spectral peaks other than characteristic peaks, produced during the EDS detection process. Examples are escape peaks and sum peaks.
Spectral Resolution	The ability to distinguish between adjacent peaks in an x-ray spectrum. It is usually determined by measuring peak width at half the maximum value of the peak height, also known as “full-width-half-maximum” (FWHM).
Sum Peak	A spectral peak occurring at twice the energy of an individual peak. Generally only seen where the element is



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	the major component and is present in high abundance.
System Dead Time	The time (expressed as a percent) that the EDS is not able to process x-rays.
System Peaks	Also known as <i>stray radiation</i> . Peaks that can occur in the x-ray spectrum resulting from interaction of the electron beam or fluorescence radiation with components of the SEM itself.
Take-off Angle	Angle between the specimen surface and the x-ray detector axis.
Trace element (EDS detection)	Trace elements, as determined from the x-ray spectrum of a sample, are those whose most intense x-ray peak is less than approximately 10% of the highest peak (relative peak height).
Working Distance	<p>The working distance is the distance between the final aperture and the specimen. Changing the working distance will have an effect on the spherical aberration of the imaging system and, therefore, will effect the resolution of the final image.</p> <p>The working distance also has an effect on the depth in which the sample appears to be in focus (depth of field).</p> <p>At a short working distance, the sample will be scanned with a wide cone of electrons resulting in an image with little depth of field. At a longer working distance, the sample will be scanned with a narrow cone of electrons resulting in an image with an increased depth of field.</p> <p>For samples having large topographical variation, a longer working distance is required to bring as much of the image into focus as possible. However, some of the resolution will be lost.</p> <p>Conversely, for relative flat samples, it is possible to obtain high resolution using a shorter working distance as there is no need for high focal depth of field.</p>



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