

# Gunshot Residue (GSR) Control Standard for Validation and Quality-Assurance Purposes

## General Purpose

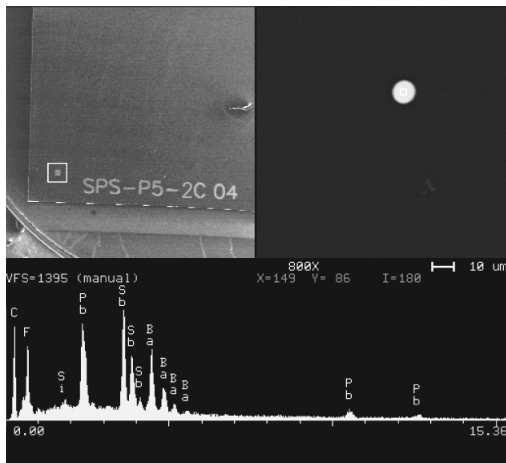
When a SEM/EDX system is used for GSR analysis it is important to achieve maximum reliability and reproducibility. A synthetic GSR calibration standard with defined Pb/Sb/Ba particles in known locations can help to avoid or detect systematic errors in the GSR analysis system and improve the quality of the overall results.

Laboratory-prepared GSR test samples can be used to monitor the reproducibility of the system. The reproducibility should be as high as possible, although it is, in most cases, below 100% owing to statistical reasons. What kinds of precautions can be taken to avoid possible systematic errors? Incorrect results are likely to occur in each performed measurement unless the operator knows the "true" values. Therefore, a standard with a precise number of particles of interest at known locations on the sample surface and with known chemical compositions, is desirable. It is almost impossible, or at least very difficult, to gain this information from "real" samples obtained by shooting and tap-lifting gunshot residue, but artificially produced samples with comparable properties can help to overcome these problems.

## Requirements

What are the desired properties for a "synthetic" test sample?

- Precisely known number of particles
- Well-defined particle diameters
- Known location of the particles
- Well-defined chemical composition of the particles



BSE image of the lower-left corner of the GSR standard  
EDX spectrum of one of the 10 $\mu$ m synthetic PbSbBa particles; The EDX spectrum shows some additional element lines for Silicon (from the substrate material and Fluorine (from the production process).

## Production

Using a patent protected production process, Pb/Sb/Ba particles are precipitated onto the surface of an 8 x 8mm silicon chip which is previously applied with a 10 $\mu$ m polyimide layer. The particles are randomly distributed but at known locations. The total number of the synthetic GSR particles, as well as their size and location on the standard, are well defined (Fig. 1).

There are four distinct particle sizes of approximately 0.5 $\mu$ m, 0.8 $\mu$ m, 1.2 $\mu$ m and 2.4 $\mu$ m in diameter. In addition, three 10 $\mu$ m particles are provided, in order to facilitate a simple cross-checking. To protect the particles from mechanical damage, the specimens are coated with a thin layer of polymer.

PbSbBa particle parameters:

- 22 particles approx.  $\phi$  0.5 $\mu$ m
- 25 particles approx.  $\phi$  0.8 $\mu$ m
- 26 particles approx.  $\phi$  1.2 $\mu$ m
- 27 particles approx.  $\phi$  2.4 $\mu$ m
- 3 particles approx.  $\phi$  10 $\mu$ m

The samples are supplied mounted on a conventional 1/2" SEM pin stub.

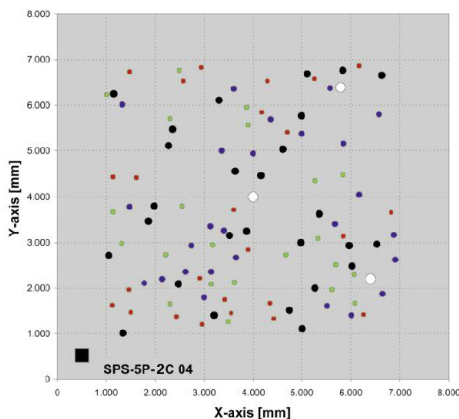


Fig. 1: Every GSR standard is supplied with a map showing the exact position of the PbSbBa particles as they were precipitated on the surface of the Silicon substrate.



Photo of GSR standard mounted on a standard 1/2" SEM pin stub.

- white: position of the 10 $\mu$ m particles
- black: position of the 2.4 $\mu$ m particles
- blue: position of the 1.2 $\mu$ m particles
- red: position of the 0.8 $\mu$ m particles
- green: position of the 0.5 $\mu$ m particles

## Results

As the exact position of each of the Pb/Sb/Ba particles is known, it is easy to evaluate the number of correctly detected particles and find explanations for missed or multiple detected particles.

Potential reasons for an incorrect number of detected particles are:

- miscalibration of the motorized stage, resulting from
  1. space between mechanical fields (missing particles)
  2. overlap of mechanical fields (multiple detected particles)
- scan hysteresis problems in the microscope column, caused by displacement of the electron beam when performing the EDX analysis,
- problems in the particle detection parameter settings (e.g., focus, dwell time, grid size in particle search, etc.) responsible for the loss of small particles.

## Benefits

Synthetic GSR standards are suitable for:

- Proficiency testing programs
- System Optimization
- System Validation
- Quality Assurance

**60806-4** SPS-5P-2 GSR Standard on 12.7mm stub (type A mount); 0.5, 0.8, 1.2 and 2.4 $\mu$ m, certified, each



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