

GSR PT - Proficiency Test on Detection and Identification of Gunshot Residue Particles by SEM/EDS

Scheme Description

In cooperation with the ENFSI expert working group Firearms/GSR



Expert Working Group
Firearms / GSR

Imprint

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Content

1	Introduction	4
1.1	Quality Standards	4
1.2	Aims of the Scheme	4
1.3	List of Abbreviations	4
2	Scheme Organisation and Management	5
2.1	Announcement	5
2.2	Website and Notification	5
2.3	Establishment of an Advisory Board	5
2.4	Timescales	6
2.5	Frequency of Participation	7
2.6	Confidentiality	7
2.7	Scheme Development	7
3	Test Material	8
3.1	Test Material Preparation	8
3.2	Quality Control	8
3.3	Distribution	8
3.4	Sample Properties	8
4	Analysis and Reporting of Results	9
4.1	Methods of Analysis	9
4.2	Performance of the Test	9
4.3	Data Reporting	10
4.4	Reporting Format	10
4.5	Late Return of Results	10
5	Performance Assessment	11
5.1	Preparation of Raw Data and Plausibility Check	11
5.2	Cross-Checking	11
5.3	Statistical Analysis	12
5.3.1	Assessment of laboratory's performance	12
5.3.2	Overall performance	13
5.3.3	Additional analyses	13
5.4	Reports and Certificates	14
5.5	Complaints	14
6	References and Sources of Information	15

1 Introduction

1.1 Quality Standards

Proficiency testing (PT) is defined by ISO/IEC 17043:2010 [1] as the use of inter-laboratory comparisons for the determination of the performance of individual laboratories in specific tests or measurements and for the monitoring of the laboratories' long-term performance.

When carried out within the context of a comprehensive quality assurance programme, proficiency testing is an independent means of reflecting the quality of test and calibration results, as described by ISO/IEC17025 [2].

All the schemes within QuoData GmbH are operated in accordance with the international guides ISO/IEC 17043:2010 and ILAC G13:2007 [3]. Furthermore, especially the forensic GSR scheme is operated in accordance with the ENFSI Guidance document [4].

1.2 Aims of the Scheme

The aim of the GSR-QS (Quality Scheme on the Detection and Identification of Gunshot Residue) is to enable laboratories to undertake forensic GSR examinations using automated SEM/EDS techniques to monitor and improve the quality of their measurements. The scheme enables laboratories to demonstrate the quality of their measurements to accreditation bodies and other appropriate authorities.

1.3 List of Abbreviations

Abbreviation	Meaning
BSE	Backscattered electrons
EDS or EDX	Energy dispersive X-ray spectroscopy
ENFSI	European Network of Forensic Science Institutes
EWG	Expert working group
GSR	Gunshot residue
PT	Proficiency test
QS	Quality scheme on the detection and identification of gunshot residue
SEM	Scanning electron microscope
TOR	Terms of reference

2 Scheme Organisation and Management

2.1 Announcement

Annually, a scheme application form is available on the QuoData website, containing information about the test materials included in the scheme, and the intended distribution dates. To participants that attended the scheme in former years an email with the announcement of the current scheme will be sent. New participants are invited to complete an application form on the QuoData website indicating their interest. However, the final decision about participation lies in the responsibility of the Advisory Board.

Additionally, the current scheme may be announced in proficiency scheme databases (e.g. EPTIS) on the internet.

2.2 Website and Notification

All deadlines will be published on the non-restricted QuoData website:

<http://quodata.de/gsr-quality-scheme.php>.

On a separate password-protected website, the final report as well as the individual result-plots and certificates can be made available for download (anonymity maintained), if necessary.

All important changes in the time scale will be announced by email as well as on the website.

2.3 Establishment of an Advisory Board

GSR-QS is managed and operated by QuoData GmbH. Technical direction and advice is provided by an Advisory Board, consisting of at least two representatives of the ENFSI Expert Working Group Firearms/GSR (ENFSI-EWG).

The members of the Advisory Board are

- Ludwig Niewöhner, PhD
Forensic Science Institute, BKA, D-65173 Wiesbaden, Germany.
- Lawrence Gunaratnam, M.Sc.
National Bureau of Investigation, NBI, PL 285, FIN-01301 Vantaa, Finland.
- Michael V. Martinez, MSFS
Bexar County Crime Laboratory, San Antonio, Texas 78229-4565, United States.

The Advisory Board may seek advice from other organisations/individuals with specific expertise on an ad hoc basis. The membership of the Advisory Board is reviewed on a regular basis.

The day-to-day operation of the scheme, including sample purchase and preparation, distribution, data processing and reporting is the responsibility of QuoData GmbH.

The terms of reference (TOR) of the Advisory Board are:

- To consider the scope and direction in which the scheme should develop.
- To represent the views of the ENFSI EWG.
- To provide specialist advice to the scheme organisers on technical and other matters, to contribute to a smooth performance of the scheme.
- To assess the results obtained in the scheme and examine the implications they have for the progress of the scheme.
- To consider the nature and timing of proficiency testing rounds and to decide on the test materials to be used.
- To assist in the revision of the scheme description.
- To advise on the promotion and publicity of the scheme.
- To provide, when requested, expert advice to participants on specific analytical difficulties encountered in the scheme.
- To discuss technical comments on each round for inclusion in the report.

The Advisory Board will meet when necessary to ensure progression of the scheme, but at least once a year.

2.4 Timescales

The scheme is operated once a year. Test materials are distributed to participants annually, with distribution dates published on the QuoData website. Samples are dispatched no later than the announced dates specified on the website. After the dispatch of the samples, laboratories have approximately 3 weeks in which to analyse the samples and report their results. Dates of the reporting deadlines are available on the QuoData website.

The structure within the scheme round is as follows:

- Procurement, preparation, dispensing and quality control testing of test materials.
- Dispatch of test materials and instructions to participants.
- Request to participants to analyse test materials and report results to QuoData GmbH as instructed and within the specified deadline.
- Data preparation and plausibility check by QuoData GmbH.
- Cross-check and possible corrections of results by laboratories.
- Analysis of results and comparison of performance of laboratories using appropriate techniques, such as z scores.
- Distribution of reports to participants.
- Review of round and identification of requirements for subsequent rounds.
- Start of next round.

Reports are issued as soon as possible after the round closure, although the timescale between closing dates and issue of final report will vary from scheme to scheme.

2.5 Frequency of Participation

As part of a comprehensive quality assurance programme, and to gain most benefit from trend analysis, an annual participation in the test is recommended.

2.6 Confidentiality

In order to ensure confidentiality, a unique laboratory reference number (Lab-ID) is allocated to each participant in all schemes. This Lab-ID enables results to be reported without divulging the identities of participant laboratories.

In cases where anonymity could not be preserved, laboratory reference numbers may be changed on request from the participating laboratory, at the discretion of QuoData GmbH.

For some schemes, participants may select to have their identity made known to others, but this will only be done with the knowledge and full permission of the participant.

2.7 Scheme Development

QuoData GmbH is continually striving to improve the test and to introduce new recommendations where appropriate. This will be accomplished in close collaboration with the Advisory Board.

3 Test Material

3.1 Test Material Preparation

Wherever practical, test materials should be as similar as possible to those routinely tested by participating laboratories. However, in some cases, in order to achieve the required degree of homogeneity and stability, test materials may be in the form of simulated samples.

In this scheme a synthetic particle sample is used with Lead/Antimony/Barium particles, which represent characteristic GSR particles. This sample shows all the criteria demanded in proficiency testing (in particular: identical sample material and homogeneity of sample sets). That means there is a certain number of synthetic GSR particles consisting of Pb, Sb and Ba on each sample and the composition of the particles as well as location and size are exactly known by the organiser.

3.2 Quality Control

Test samples are, as far as possible, prepared using a well-controlled process, which has been verified to produce homogeneous materials. If, in the opinion of QuoData GmbH, any material does not meet homogeneity requirements, a replacement material will be obtained for dispatch. Details of tests performed, acceptability criteria and results will be given in the scheme reports.

3.3 Distribution

Test materials are sent in appropriate packaging and under conditions chosen to protect the samples during transit.

Participants are asked to check the contents of packages immediately after reception and to contact QuoData GmbH if there are any problems with the condition of the test materials or accompanying documents.

3.4 Sample Properties

A glassy carbon chip of 8 x 8 mm² is mounted on a standard 1/2-inch stub. On this chip there is an area of 6 x 6 mm² where an exactly defined number of PbSbBa particles is distributed (the composition of the "GSR particles" is Pb, Sb, Ba, and F; the F-signal results from the BaF₂ that is used within the sample preparation process). The PbSbBa particles have to be searched and filed.

The sample is almost free of "contamination" for reasons of a standard for system validation purposes in future applications. For protective reasons the chip has finally been coated with a thin carbon layer, which is supposed to avoid charging. Nevertheless, if charging occurs, the participants are requested to perform a supplementary carbon coating of the sample.

4 Analysis and Reporting of Results

4.1 Methods of Analysis

Participants are asked to treat the PT material in the same way as a routine sample. The analysis of the test sample should be performed – where possible – with the same SEM/EDS parameter settings as used for routine casework.

Participants are requested to report also their acquisition parameters. It is important that this information is accurate as the results are analysed and reported according to the parameters stated.

4.2 Performance of the Test

The test sample has to be mounted on the stage in such a way that the small 100 x 100 μm^2 pad is displayed in the lower left corner of the SEM screen (see Figure 1). At least the centre area of 6 x 6 mm^2 of the chip which is margined by four markers needs to be examined.

If the BSE threshold adjustment has to be changed compared to the participant's standard settings, it is recommended to use the 100 x 100 μm^2 pad or the 10 μm PbSbBa particle, the latter located exactly in the centre of the chip, for a suitable BSE adjustment. Particle sizes cover the range between sub- μm and several μm in diameter.

Due to the production process the particles also contain some amount of Fluorine. However, if an interfering F-signal in the obtained spectra is leading to a false classification of the PbSbBa particles, it is suggested to either add Fluorine as a matrix element in your criteria list or set the F-signal to zero. Any of these necessary changes of the standard settings should be noted as a comment in the answering form. If there are particles PbSb, PbBa or SbBa put into the particle classification scheme, these should be reported as well.

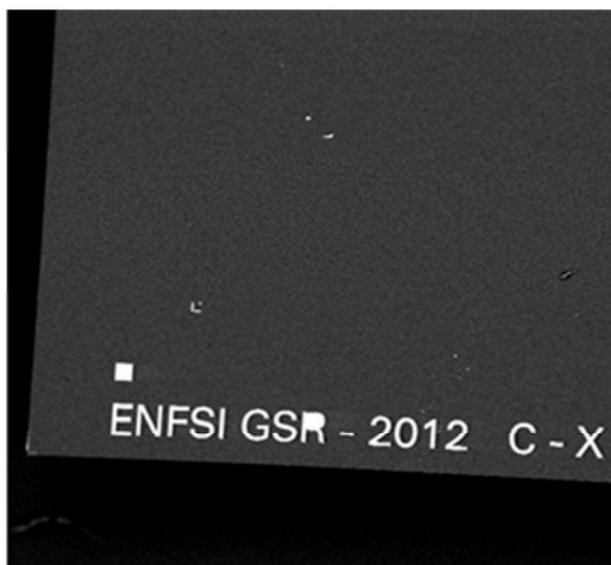


Figure 1: BSE image of the lower left corner part of a GSR test sample

4.3 Data Reporting

The raw particle data obtained by means of the measurement have to be submitted to QuoData GmbH. For this purpose a web-based answering form is available on a separate password-protected QuoData website.

This website consists of two forms, both needed to be filled out. The first form lists questions concerning the instrumental and procedural conditions of your measurements (SEM/EDS system data and some SEM/EDS acquisition parameters). In the second form the raw particle data can be uploaded or pasted. The raw particle data can also be submitted in paper form. There is an additional charge by QuoData GmbH to enter data from paper form.

4.4 Reporting Format

The raw particle data should contain at least the following information:

- absolute X coordinate (in mm or μm),
- absolute Y-coordinate (in mm or μm),
- calculated particle diameter (μm) and
- classification of the particle.

Results received after the deadline for any particular round will only be included under exceptional circumstances and in agreement with QuoData GmbH and the Advisory Board.

It is recommended that results are checked thoroughly before reporting. Once submitted and received, results may only be amended at the discretion of the scheme coordinator.

No changes can be made after the assigned values have been reported. Results should be reported clearly, in the form and units requested. Results entered incorrectly will not be edited by QuoData GmbH.

4.5 Late Return of Results

Participants are asked to return results by the given deadline in order to ensure that their results are included in the statistical analysis and the scheme report. Results received after the closure date can be excluded from the overall assessment and can remain unconsidered in the report.

5 Performance Assessment

5.1 Preparation of Raw Data and Plausibility Check

Within 15 working days after the deadline for submitting results, QuoData GmbH will prepare the data and carry out plausibility checks. Thereby, the reported data of each laboratory will be transferred into individual result plots (see Figure 2). Such a plot displays all correctly detected 'regular' PbSbBa-particles in an XY-plot and enables the laboratory to check their submitted results.

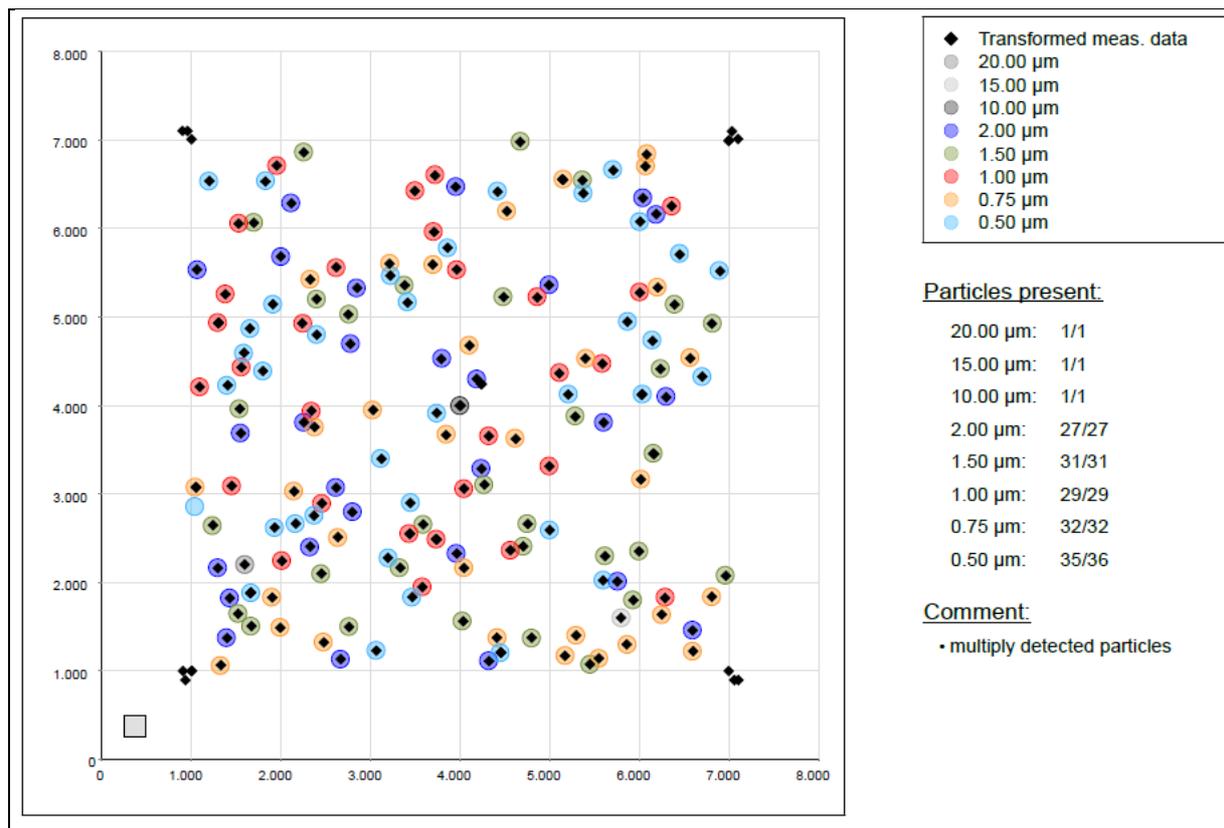


Figure 2: Individual result plot

5.2 Cross-Checking

After the individual result plots have been sent to the laboratories by email (hard copy versions may be dispatched as well, but additional charge applies), laboratories have the possibility to cross-check their results. Corrections or comments need to be made within 10 working days after the individual result plots have been sent. The deadline for potential corrections will be released on the QuoData website in time.

5.3 Statistical Analysis

5.3.1 Assessment of laboratory's performance

5.3.1.1 On the basis of the detection capability

For each laboratory the 90 % detection capability, i.e. the particle size of PbSbBa-particles which the laboratory detects and identifies correctly, is calculated and assessed. The 90 % detection capability arises from the detection capability curve which describes the probability of correct detection as function of the particle size. It is assumed that the detection capability curve follows a 3-parameter sigmoid curve. The three parameters are estimated using Maximum Likelihood Analysis. The uncertainty of the detection capability curve is determined applying a parametric Bootstrap method.

The laboratory result of the 90 % detection capability is assessed using z scores according to ISO and EURACHEM ([5], [6]). The z score compares the difference between the participant's result (x_p) and the assigned value (x_a) in terms of the acceptable spread of results or standard deviation for proficiency assessment (σ_{pt}):

$$z = \frac{x_p - x_a}{\sigma_{pt}} .$$

The assigned value is the mean performance of all laboratories that participated in this proficiency test. The standard deviation for proficiency assessment (σ_{pt}) results from the standard uncertainty of the assigned value (σ_a) and the reproducibility standard deviation (σ_r) across all laboratory-specific values of the 90 % detection capability (σ_e) by the following calculation: $\sigma_t = \sqrt{\sigma_a^2 + \sigma_r^2}$.

The reproducibility standard deviation¹ is calculated by the robust statistical "Q-method" ([6], [7], [8]) according to ISO 13528 [6]. For the performance assessment, the following classification is assumed:

Satisfactory result:	$z \geq -2$
Questionable result:	$-3 \leq z < -2$
Unsatisfactory result:	$z < -3$

The assessment is performed using the software package PROLab Plus 2015 [9].

5.3.1.2 On the basis of the number of correctly detected PbSbBa-particles

For information purposes only, the individual performance is also assessed on the basis of the number of correctly detected PbSbBa-particles. For each particle size evaluated and each laboratory a z score is calculated. As assigned value, the true value for the number of PbSbBa-particles is used. The standard deviation for proficiency assessment is set to the minimum of 10 % of the assigned value and the reproducibility standard deviation. The reproducibility standard deviation is calculated by the statistical "Q-method" according to ISO 13528:2015. This robust method is selected in order to take into account the discrete nature of the number of correctly detected PbSbBa-particles and to minimize the effect of potential outliers.

¹ The reproducibility standard deviation characterizes the variability of the measurement data under reproducibility conditions, i.e. test results are obtained with the same method on identical test items in different laboratories with different laboratory assistants using different laboratory equipment.

For the performance assessment, the following classification is assumed:

Satisfactory result:		$ z \leq 2.0$
Questionable result:	$2.0 <$	$ z \leq 3.0$
Unsatisfactory result:	$3.0 <$	$ z $

5.3.2 Overall performance

The overall performance is displayed by the overall detection capability curve. In order to quantify the overall detection capability, the 3-parameter model is fitted to the mean across all laboratories that participated in the proficiency test.

The running scheme of the proficiency test allows the comparison of the method detection capability of the current proficiency test to the method detection capabilities obtained in former tests.

5.3.3 Additional analyses

5.3.3.1 Correlation of laboratory performance within two proficiency tests

Additionally, the running scheme of the proficiency test allows for a comparison of the obtained results for those laboratories that participated in the same scheme frequently. Therefore the z scores obtained in two proficiency tests are shown for the laboratories (evaluation according to Youden [10]). Thereby it is possible to see whether a laboratory shows a consistently satisfactory performance, if the performance improved or if a change for the worse has to be observed, and finally if the performance continues to be unsatisfactory.

5.3.3.2 Other additional analyses

Additional statistical analyses may be carried out if necessary or if suggested by the Advisory Board.

5.4 Reports and Certificates

Reports will be sent to participants by email within 60 working days from the cross checking deadline. If requested, hard copy reports may be dispatched as well (additional charge applies).

Participant results will only be identified by the Lab-ID and the instrument used by each participant will not be reported. However, to assist instrument manufacturers with their future development and with their advisory capacity, summary statistics for each instrument will be provided to the appropriate instrument manufacturer. This information will not contain any individual participant results, thereby maintaining confidentiality. If participants have a poor performance score and wish to seek advice from the appropriate instrument manufacturers it is their responsibility to contact them via QuoData GmbH or using the contact details provided in the report.

Individual certificates for each participant including the obtained z scores and the laboratory detection capability curve are provided with the reports.

The report is made available on the website.

5.5 Complaints

In case of complaints, these will be fully investigated according to our quality management system to determine the underlying cause and to decide upon a course of action. This course of action together with results of any investigations carried out will be communicated to the participant.

6 References and Sources of Information

- [1] ISO/IEC 17043:2010: '*Conformity assessment – General requirements for proficiency testing*'.
- [2] ISO/IEC 17025:2005: '*General requirements for the competence of testing and calibration laboratories*'.
- [3] ILAC (International Laboratory Accreditation Cooperation) G13:2007: '*Guide to the Requirements for the Competence of Providers of Proficiency Testing Schemes*'.
- [4] ENFSI Standing Committee for Quality and Competence (QCC): '*Guidance on the Conduct of Proficiency Tests and Collaborative Exercises Within ENFSI*'; 27-06-2014.
- [5] EURACHEM Guide on "Selection, Use and Interpretation of Proficiency Testing (PT) Schemes by Laboratories"; Second Edition 2011;
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