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GSR PT – Proficiency Test on Detection and Identification of Gunshot Residue Particles by SEM/EDS according to ASTM E1588

Scheme Description

In cooperation with the ENFSI expert working group Firearms/GSR

ENFSI
Expert Working Group
Firearms / GSR

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1 Introduction

1.1 Quality Standards

Proficiency testing (PT) is defined by ISO/IEC 17043:2010 [1] as the use of interlaboratory 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/IEC 17025 [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 according to ASTM E1588 [5], 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. This scheme is designed for 50 to 1000 expected participants annually.

1.3 List of Abbreviations

Abbreviation	Meaning
BSE	Backscattered electrons
EDS	Energy dispersive X-ray spectroscopy (also known as EDX)
ENFSI	European Network of Forensic Science Institutes
EWG	Expert working group
GSR	Gunshot residue
PT	Proficiency test
QC	Quality control
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 website of the QuoData GmbH (as given in Section 2.2), 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 website of the QuoData GmbH, 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 website of the QuoData GmbH:

https://quodata.de/de/ringversuche/gsr-quality-scheme.html

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

2.3 GSR PT Online Portal

Since 2019, the GSR profiency tests are managed via the GSR PT Online Portal available at https://gsr.quodata.de.

Each participant has a separate account, where the data reported as well as the final reports of results, the individual results plots as well as the individual certificates of the respective participant's institution are made available. Access to the documents is possible at any time – for all PT rounds participated in from 2019.

The enrollment for a GSR PT round is also done via the GSR PT Online Portal – so far the interesting participant is already registered.

The registration for the GSR PT Online Portal is possible via an online application form provided here:

https://gsr.quodata.de/pt-participant/lab_applicant/add

2.4 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, 65193 Wiesbaden, Germany.
- Amalia Brouwer-Stamouli, PhD
  Netherlands Forensic Institute, NFI, 2490 AA The Hague, Netherlands.

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.

QuoData GmbH is responsible for the planning of the proficiency test scheme, the evaluation of performance, and the authorization of the final report, according to ISO 17043. In addition, QuoData GmbH manages also the day-to-day operation of the scheme, including sample purchase and preparation, dispatch, data processing, reporting of the results in a final report, and providing individual certificates.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.

2.5 Timescales

The scheme is operated once a year. Test materials are distributed to participants annually, with distribution dates published on the website of the QuoData GmbH[1]. Samples are dispatched no later than the announced dates specified on the website. After the dispatch of the samples, laboratories have approximately four weeks in which to analyse the samples and report their results.

Dates of the reporting deadlines are also available on the website of the QuoData GmbH[1].

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 final report of results and individual certificates to participants.
- Review of PT round and identification of requirements for subsequent PT rounds.
- Start of the subsequent PT round.

The final report of results is issued as soon as possible after the round closure, although the timescale between closing dates and issue of final report of results can vary from round to round.

All important changes in the timescale will be announced by email as well as on the website of the QuoData GmbH[1].

[1] https://quodata.de/de/ringversuche/gsr-quality-scheme.html

2.6 Frequency of Participation

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

2.7 Confidentiality

In order to ensure confidentiality, a unique laboratory reference number (Lab ID) is allocated to each participant in all PT rounds, where the Lab IDs differ from round to round per participant. 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 PT rounds, 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.

2.8 Subcontracting services

Various aspects of the GSR PT scheme can from time to time be subcontracted. When subcontracting occurs, it is placed with a competent subcontractor and the proficiency testing provider is responsible for this work.

2.9 Scheme Development

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

2.10 Potential major sources of errors

The potential major sources of errors involved in the area of proficiency testing are offered in Table 1.

Table 1: Potential major sources of errors in the GSR PT program

No.	Phase	Label	Potential error in each phase
1	Preparation of samples	a	varying of coating parameters
		b	errors in the data conversion for the mask production
		c	missing particles
2	Dispatch of test samples to the participants	a	defect or damage of the test samples during the transport
3	Measuring errors at the participants' site	a	wrong alignment of the sample on the SEM/EDS
		b	unsuitable choice of parameters during the measurement process
		c	no usage of the Standard Operating Procedure (SOP) for measuring the sample
4	Submission of data by the participants	a	submission of incomplete data sets
5	Data preparation and plausibility check	a	errors in the data submission process by the participants
6	Generation of the final report of results	a	wrong transfer of the data (tables, graphics) into the final report of results
7	Generation of the individual certificates	a	wrong transfer of the laboratory specific results into their certificates

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 material. 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

The test material is sent in an 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 BaF_2 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 with a automated software control [5].

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 µm² pad is displayed in the lower left corner of the SEM screen (see e.g. Figure 1). At least the centre area of 6 x 6 mm² 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 µm² 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.

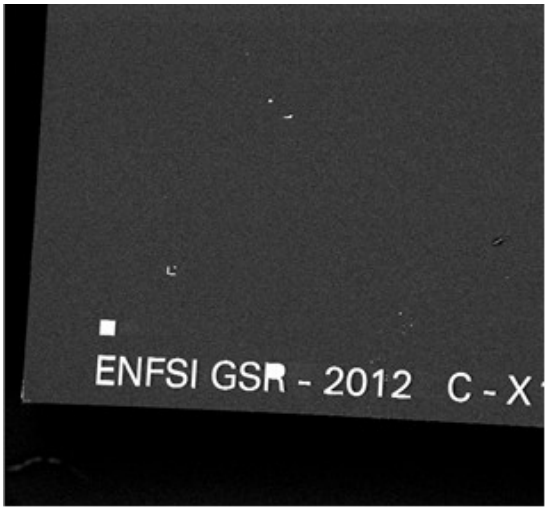

Figure 1: BSE image of the lower left corner part of a GSR test sample

4.3 Data Reporting

Sample results are reported to QuoData GmbH via a web-based online data entry form, which is available on the GSR PT Online Portal (https://gsr.quodata.de/).

In this online data entry form, the raw particle data are uploaded or pasted as well as additional information on instrumental and procedural conditions of the participant's measurements (i.e. SEM/EDS system data and some SEM/EDS acquisition parameters) are entered.

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 if the cross-check period has expired. Results should be reported clearly, in the format requested. Incorrect entered results will not be edited by QuoData GmbH.

4.5 Late Return of Results

Participants are asked to return results within the given deadline to ensure that their results are included in the statistical analysis and also in the final report of results. Results received after the closure date may be excluded from the overall assessment and disregarded in final report of results. An individual certificate will however still be issued.

5 Performance Assessment

5.1 Preparation of Raw Data and Plausibility Check

Within 15 working days after the deadline for submitting test 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 e.g. 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.

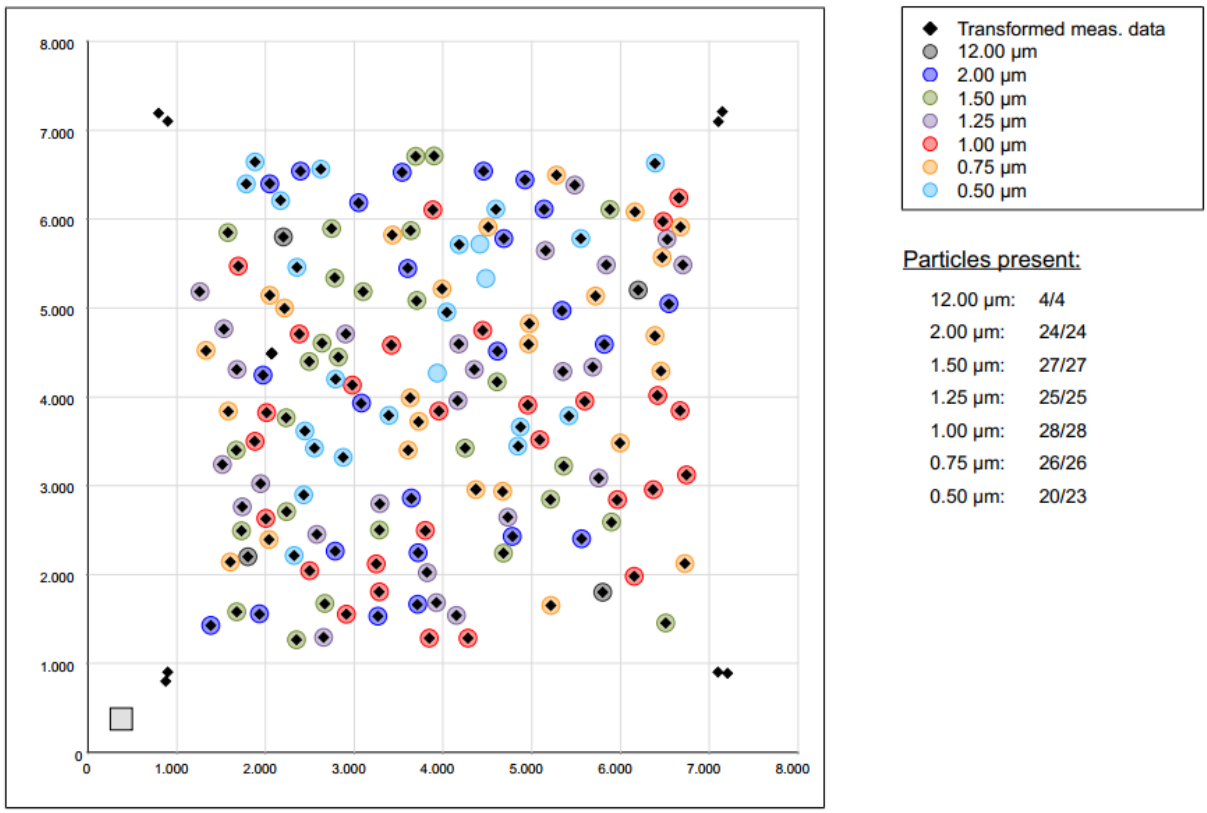

Figure 2: Individual result plot

5.2 Cross-Checking

After the individual result plots have been made available to the laboratories in the GSR PT Online Portal[2] (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 ten working days after the individual result plots have been made available. The deadline for potential corrections will be released on the website of the QuoData GmbH in time.

[2] https://gsr.quodata.de

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 13528:2015 [6] and EURACHEM [7]. 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 x_a is the mean 90 % detection capability across 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 by the following calculation: $\sigma_{pt}=\sqrt{{\sigma_a}^2+{\sigma_R}^2}$.

The reproducibility standard deviation[3] σ_R is calculated by the robust statistical Q method ([6], [8], [9]) according to ISO 13528:2015 [6]. For the performance assessment, the following classification is assumed:

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

The assessment is performed using the current version of the software package PROLab Plus [10].

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 x_a, the true value for the number of PbSbBa-particles is used. The standard deviation for proficiency assessment σ_{pt} is set to the minimum of 10 % of the assigned value

[3] The reproducibility standard deviation σ_R 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.

and the reproducibility standard deviation σ_R. The reproducibility standard deviation σ_R is calculated by the robust statistical Q method ([6], [8], [9]). 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.

For the performance assessment, the following classification is assumed:

Satisfactory result: $-2.0 \leq z \leq 0.0$
Questionable result: $-3.0 < z < -2.0$
Unsatisfactory result: $z \leq -3.0$

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 sigmoid curve is fitted to the mean across all laboratories that participated in the current PT round.

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 PT rounds.

5.3.3 Additional analyses

5.3.3.1 Correlation of laboratory performance between two PT rounds

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 subsequent PT rounds (the current PT round compared to the previous PT round) are shown for the laboratories (evaluation according to Youden [11]). 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 Bias analysis

It is possible to perform a further analysis of overall laboratory performance by breaking down observed errors into different components. Three bias components will be considered:

- **Detection bias**: This *negative* bias component corresponds to failure to detect a PbSbBa particle
- **Classification bias**: This *negative* bias component corresponds to failure to correctly classify a previously detected PbSbBa particle as characteristic or consistent
- **Double-count bias**: This *positive* bias component corresponds to an instance of a PbSbBa particle being detected and correctly classified more than once. In practice, no more than one double-count per PbSbBa particle is observed. However, in theory, nothing prevents one PbSbBa particle from being detected more than twice. If one and the same PbSbBa particle were detected three times, for instance, this would represent one correct detection and two double-counts.

This bias analysis is provided for information purposes only and may become an integral part of future assessements.

5.3.3.3 Other additional analyses

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

5.4 Final Report of Results and Individual Certificates

The final report of results and the individual certificates will be published electronically to participants within a maximum of 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.

Individual certificates for each participant including the the laboratory detection capability curve and obtained z scores are provided with the final report of results.

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.

In case of any concerns regarding to the correct conduct or evaluation of the PT round or about the assessment of the participant's proficiency, there is the opportunity to appeal within one month after publishing the final report by contacting GSR-ENFSI-PT@quodata.de.

6 References and Sources of Information

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