

GSR2011

**PROFICIENCY TEST ON DETECTION AND
IDENTIFICATION OF GUNSHOT RESIDUE
PARTICLES BY SEM/EDX**

Scheme Description



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Contents

1	INTRODUCTION	4
1.1	Quality Standards	4
1.2	Aims of the Scheme	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	6
2.6	Confidentiality	6
2.7	Product development	6
3	TEST MATERIAL	7
3.1	Test Material Preparation	7
3.2	Quality Control	7
3.3	Distribution	7
3.4	Sample Properties	7
4	ANALYSIS AND REPORTING OF RESULTS	8
4.1	Methods of Analysis	8
4.2	Performance of the Test	8
4.3	Reporting Format	9
4.4	Late Return of Results	9
5	PERFORMANCE ASSESSMENT	10
5.1	Preparation of Raw Data and Plausibility Check	10
5.2	Cross-Checking	10
5.3	Statistical Analysis	10
5.3.1	Method and Laboratory Assessment	10
5.3.2	Method and Individual Laboratory Detection Capability	11
5.3.3	Youden Plots	11
5.3.4	Additional Analyses	11
5.4	Reports and Certificates	12
5.5	Complaints	12
6	APPENDIX	13
6.1	References and Sources of Information	13
6.2	Members of the Advisory Board	14
6.3	List of abbreviations	14
6.4	Reporting format for GSR-QS test	14

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 quo data 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]. If demanded, quo data GmbH is prepared to go for an accreditation e.g. by the DAP Accreditation Service for the provision of proficiency testing schemes [5].

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/EDX 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.

2 SCHEME ORGANISATION AND MANAGEMENT

2.1 Announcement

Annually, a scheme application form is available on the quo data 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 quo data 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 databases (e.g. EPTIS) on the internet.

2.2 Website and Notification

All deadlines will be published on the non-restricted quo data website. On a separate password-protected website, the final report as well as the individual result-plots and certificates will be available for download (anonymity maintained).

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 quo data 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 listed in Appendix 6.2. 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 quo data 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 quo data 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 quo data 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 quo data GmbH as instructed and within the specified deadline.
- Data preparation and plausibility check by quo data 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 quo data 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 Product development

quo data 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 quo data 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 quo data GmbH if there are any problems with the condition of the test materials or accompanying documents.

3.4 Sample Properties

A silicon chip of 8 x 8 mm² is mounted on a standard 1/2-inch stub. On this chip there is an area of 7 x 7 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 was used within the sample preparation process). The PbSbBa particles have to be searched for and filed.

The sample is almost free of "contamination" for reasons of a standard for fast verification 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/EDX 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 upper left corner of the SEM screen (see Figure 1). At least the centre area of 7 x 7 mm^2 of the chip 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 a 10 μm thick carbon coating as intermediate layer between the silicon substrate and the particles, no disturbing silicon line in the EDX spectrum should occur.

However, if an interfering Si-signal in the obtained spectra is leading to a false classification of the PbSbBa particles, it is recommended to either add Silicon as a matrix element in the criteria list of the software or set the Si-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.

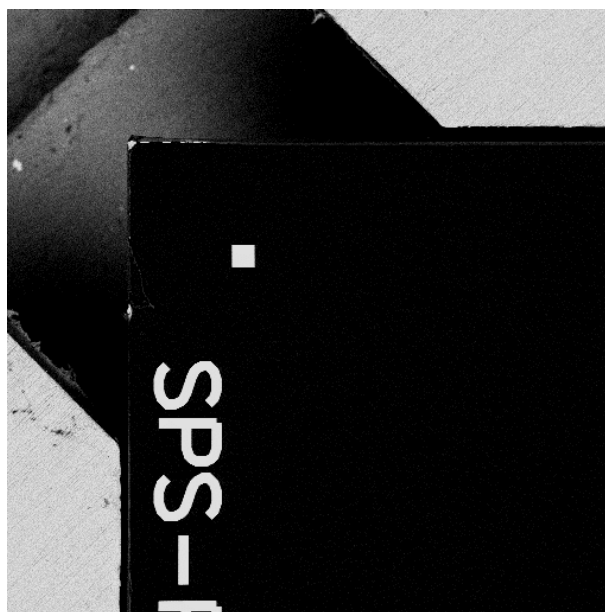


Figure 1: BSE image of the upper left corner part of the test sample

4.3 Reporting Format

Results are to be submitted to quo data GmbH either electronically, using the “GSR-Report template” on the quo data website, or in paper form. There is an additional charge by quo data GmbH to enter data from paper form.

Results received after the deadline for any particular round will only be included under exceptional circumstances and in agreement with quo data 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.

The reporting format for the GSR-QS test is described in detail in Appendix 6.4. Results entered incorrectly will not be edited by quo data GmbH.

4.4 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 will not be included 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, quo data 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). These plots enable the laboratories to check their submitted results.

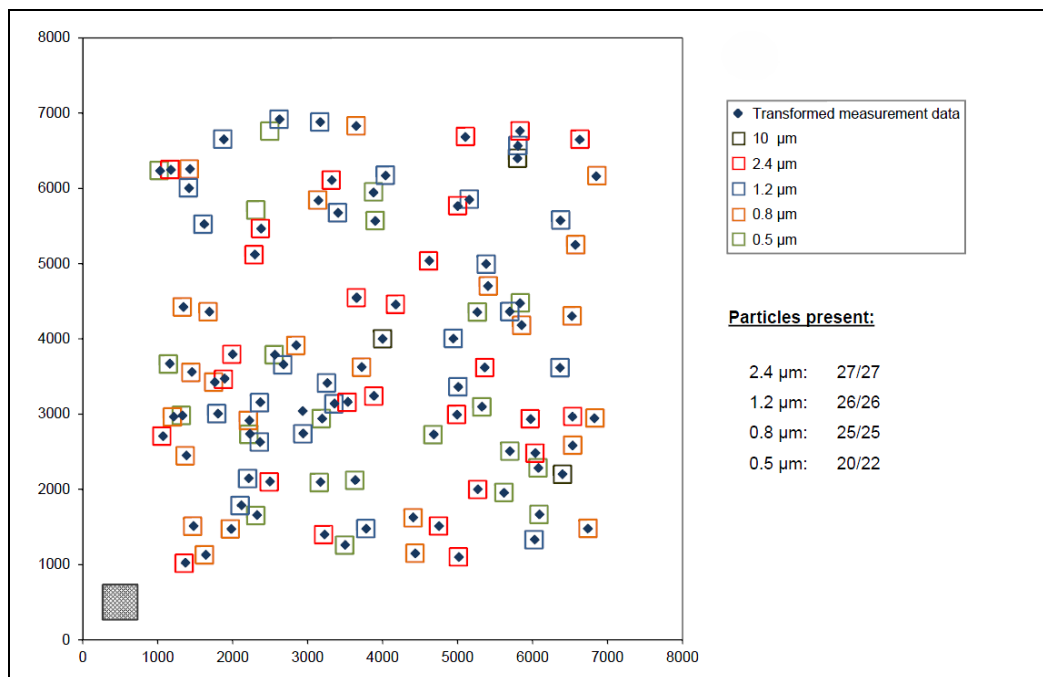


Figure 2: Individual result plot

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 quo data website in time.

The individual result plots will be made available on the password protected website with anonymity maintained.

5.3 Statistical Analysis

5.3.1 Method and Laboratory Assessment

All results are collated and the data are then analysed. The assessment is performed using the software package ProLab Plus 2010 [6], which is widely employed for the evaluation of methods interlaboratory tests and laboratory proficiency tests. The robust statistical methods according to DIN 38402 A45 (Hampel estimator and Q-method, [7], [8], [9]) are applied in order to take into account the discrete nature of the data.

For laboratory assessment, Z scores are employed. Z scores are performance scores, which compare the difference between each participant's result and the assigned value in terms of the acceptable spread of results or standard deviation for proficiency assessment. Therefore, assigned value and target standard deviation are necessary. There are several recognised ways to establish the assigned value in a proficiency testing scheme. In GSR-QS the true number of PbSbBa-particles for the individual particle sizes is known, in order to allow an error of 1 particle (e.g. due to insufficient homogeneity), the assigned value is set to one less than the true value. For the relative target standard deviation (s.d.) the empirical standard deviation is assumed, however, an upper limit of 10% of the assigned value is set.

Once the assigned value and the target standard deviation have been determined, a Z score for each laboratory is then calculated:

$$Z = \frac{\text{laboratory's result} - \text{assigned value}}{\text{target standard deviation}}.$$

For the purposes of performance assessment, the following classification is assumed:

Satisfactory result:		Z	≤	2.00	
Questionable result:	2.00	<	Z	≤	3.00
Unsatisfactory result:	3.00	<	Z		

5.3.2 Method and Individual Laboratory Detection Capability

Method (overall) and individual laboratory detection capability curves are determined. For the overall detection capability, the 95%-confidence interval is determined applying a parametric Bootstrap method.

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 Youden Plots

Additionally, the running scheme of the proficiency test allows a comparison of the obtained results for those laboratories that participated in the same scheme frequently. Therefore a certain feature, e.g. a distinct particle size class is chosen and the performances within both proficiency tests are shown for the given 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.

5.3.4 Additional Analyses

Additional statistical analyses may be carried out if necessary or if suggested by the Advisory Board. This may comprise a comparison of the method detection capability of different methods or laboratory-specific analyses, e.g. determining the so-called running Z scores (over several years of participation).

5.4 Reports and Certificates

Reports will be sent to participants by email within 30 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 quo data 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 as well as the certificates are made available on a password-protected website.

5.5 Complaints

In case of complaints, these will be fully investigated according to our quality 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 APPENDIX

6.1 References and Sources of Information

- [1] EN ISO/IEC 17043:2010: '*Conformity assessment – General requirements for proficiency testing*'; 2010.
- [2] BS EN ISO/IEC 17025: '*General requirements for the competence of testing and calibration laboratories*'; 2005.
- [3] ILAC (International Laboratory Accreditation Cooperation) G13: '*Guide to the Requirements for the Competence of Providers of Proficiency Testing Schemes*'; 2007.
- [4] Guidance on the Conduct of Proficiency Tests and Collaborative Exercises Within ENFSI; Ref. code: QCC-PT-001; Issue No.: 003; 18-02-2005.
- [5] DAP (Deutsches Akkreditierungssystem Prüfwesen GmbH); www.dap.de/profile.html.
- [6] ProLab Plus 2010; quo data GmbH; Dresden, Germany.
- [7] Huber P.J.; '*Robust statistics*'; John Wiley & Sons; New York; 1981.
- [8] Rousseeuw P.J.; '*Tutorial to Robust Statistics*'; Journal of Chemometrics 5; pp.1-20; 1991.
- [9] DIN 38402 – A45: '*Interlaboratory Comparisons for Proficiency Testing of Laboratories*'; 2003.
- [10] Youden W.J.; '*Statistical techniques for collaborative tests*'; Statistical manual of the OAC; Gaithersburg: AOAC International; 1975.
- [11] Niewöhner L., Wenz W., Andrasko J., Beijer R., Gunaratnam L.; '*ENFSI Proficiency Test Program on Identification of GSR by SEM/EDX*'; Journal of Forensic Sciences; vol. 48(4); (2003); pp. 786-792.
- [12] Niewöhner L., Andrasko J., Biegstraaten J., Gunaratnam L., Steffen S., Uhlig S.; '*Maintenance of the ENFSI Proficiency Test Program on Identification of GSR by SEM/EDX (GSR2003)*'; Journal of Forensic Sciences; vol. 50(4); (2005).
- [13] Niewöhner L., Andrasko J., Biegstraaten J., Gunaratnam L., Steffen S., Uhlig S., Antoni, S.; '*GSR2005-Continuity of the ENFSI Proficiency Test on Identification of GSR by SEM/EDX*'; Journal of Forensic Sciences; vol. 53(1); (2008).

6.2 Members of the Advisory Board

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6.3 List of abbreviations

Abbreviation	Meaning
BSE	backscattered electrons
ENFSI-EWG	European Network of Forensic Science Institutes – expert working group
EDX	energy dispersive X-ray spectroscopy
GSR	gunshot residue
PT	proficiency testing
QS	quality scheme
SEM	scanning electron microscope
TOR	terms of reference

6.4 Reporting format for GSR-QS test

The results of the test have to be sent to quo data GmbH electronically using the template on the quo data website. If sent in an EXCEL spreadsheet or in paper form, at least the following columns should be obtained:

- particle no.,
- absolute X coordinate (in mm or μm),
- absolute Y-coordinate (in mm or μm),
- calculated particle diameter (μm) and
- classification of the particle.

Additionally, a form containing SEM/EDX system data and some SEM/EDX acquisition parameters has to be filled in (see e. g. *Answering Form* for the GSR2010 test).