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International Proficiency Testing Scheme for the Detection and Classification of Gunshot Residues by SEM/EDS

In cooperation with the ENFSI Expert Working Group Firearms/GSR

Scheme Description

ENFSI
Expert Working Group
Firearms / GSR

DAkkS
Deutsche
Akkreditierungsstelle
D-EP-21563-01-00

Provider of the proficiency testing scheme

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Proficiency testing provider accredited by DAkkS according to DIN EN ISO/IEC 17043:2010.

The accreditation is valid only for the scope listed in the annex of the accreditation certificate D-EP-21563-01-00.

The DAkkS is signatory of the multilateral arrangements of EA, ILAC and IAF for mutual recognition.

DAkkS Deutsche Akkreditierungsstelle D-EP-21563-01-00

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January 2024

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Contents

List of abbreviations

Abbreviation	Meaning
BSE	Backscattered electrons
DAkkS	Deutsche Akkreditierungsstelle GmbH
EDS	Energy dispersive X-ray spectroscopy (also known as EDX)
ENFSI	European Network of Forensic Science Institutes
GSR	Gunshot residue
PT	Proficiency test
QC	Quality control
SEM	Scanning electron microscope
TOR	Terms of reference

1 Introduction

1.1 Quality standards

Proficiency testing (PT) is defined by DIN EN 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 DIN EN ISO/IEC 17025:2018 [2].

The GSR PT scheme for the Detection and Classification of Gunshot Residue Particles by SEM/EDS according to ASTM E1588 [5] provided by quo data Gesellschaft für Qualitätsmanagement und Statistik mbH (hereafter referred to as QuoData GmbH) is operated in accordance with the international guides DIN ISO/IEC 17043:2010 [1] and ILAC G13:2007 [3] as well as with the ENFSI Guidance document [4].

Since April 2022, the GSR PT scheme is accredited by DAkkS according to DIN EN ISO/IEC 17043:2010 [1].

1.2 Aims of the GSR PT scheme

The aim of the GSR PT scheme is to enable laboratories to perform forensic GSR examinations using automated SEM/EDS techniques according to ASTM E1588-20 [5], to monitor and to improve the quality of their measurements. The GSR PT scheme enables laboratories to demonstrate the quality of their measurements to accreditation bodies and other appropriate authorities.

Based on specially prepared test samples with synthetic lead/antimony/barium particles, which represent characteristic GSR particles as realistic as possible, it is checked whether the participating laboratories are able to correctly detect and classify all available particles by applying their own laboratory-specific method/SOP for the *automated* detection and classification of synthetic GSR particles by SEM/EDS. Particles detected and/or classified using manual procedures are not considered for statistical evaluation and assessment of laboratories' performance.

The GSR PT scheme is designed for 50 to 1000 expected participants annually.

2 GSR PT scheme: Organisation and management

2.1 Announcement

A GSR PT scheme description and information on the current or upcoming PT round is available on the website of the QuoData GmbH (as given in Section 2.2), containing information about the test materials included in the GSR PT scheme, and the intended distribution dates. To participants that attended the GSR PT scheme in former years, an email with the announcement of the current PT round will be sent. New participants are invited to complete an application form on the GSR PT portal provided by the QuoData GmbH, indicating their interest. State-registered forensic science laboratories and all members of the European Network of Forensic Science Institutes (ENFSI) are welcome to participate. However, the final decision about participation lies in the responsibility of the advisory board.

Additionally, the current PT round may be announced in proficiency testing 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/gsr-quality-scheme.php

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

2.3 GSR PT portal

Since 2019, the GSR PT scheme is managed via the GSR PT portal available at

https://forensics-pt.quodata.de.

Each participant has an individual account, where the reported data, 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 onwards.

The enrollment for a GSR PT round is also done via the GSR PT portal.

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

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

2.4 Establishment of the Advisory Board

The GSR PT scheme is is organized, conducted and evaluated by QuoData GmbH. Technical direction and advice is provided by the Advisory Board, consisting of at least two representatives of the ENFSI Expert Working Group Firearms/GSR.

The current members of the Advisory Board are

- Ludwig Niewöhner, Ph.D.
  Forensic Science Institute, BKA, 65193 Wiesbaden, Germany.
- Glenn R.C. Roepnarain, Bsc.
  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 and coordination of the GSR PT scheme, the evaluation of performance, and the authorization of the final report, according to DIN EN ISO/IEC 17043:2010 [1]. In addition, QuoData GmbH manages also the day-to-day operation of the PT rounds, 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 GSR PT scheme should develop.
- To represent the views of the ENFSI Expert Working Group Firearms/GSR.
- To provide specialist advice to the GSR PT scheme organisers on technical and other matters, to contribute to a smooth performance of the GSR PT scheme.
- To assess the results obtained in the GSR PT scheme and examine the implications they have for the progress of the GSR PT 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 GSR PT scheme description.
- To advise on the promotion and publicity of the GSR PT scheme.
- To provide, when requested, expert advice to participants on specific analytical difficulties encountered in the GSR PT 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 GSR PT scheme, but at least once a year.

2.5 Timeline

The GSR PT 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 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 a PT 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 (within a maximum of 60 working days from the cross checking deadline), although the time span between round closure and issuing of the final report of results can vary from round to round.

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

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

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 GSR PT 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. All participants are named in the reports with their anonymized lab code only, and data of individual participants may only be passed on with the prior consent of the participant. However, the participants agree that the proficiency testing provider or the Advisory Board may publish the results in anonymized form.

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

The final reports of results and all results pertaining to the specific proficiency test rounds must be treated as confidential unless they are presented and discussed at the annual meeting of the ENFSI Expert Working Group Firearms/GSR and/or published by the Advisory Board. The use of final reports of results is permitted to the participants exclusively for internal purposes and within the framework of legal requirements.

If participating laboratories intend to disclose their laboratory code for some PT rounds or to reproduce a final report of results, even in part, the approval of the proficiency testing provider and the Advisory Board must be obtained.

2.8 Subcontracting services

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

2.9 GSR PT scheme development

QuoData GmbH is continually striving to improve the GSR PT scheme 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 shown in Table 1.

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

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 Preparation of test material

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 GSR PT scheme, a synthetic particle sample with lead/antimony/barium particles, which represent characteristic GSR particles, is used. This sample shows all the criteria demanded in proficiency testing (in particular: identical sample material and homogeneity of sample sets). That means that 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 the location and size are exactly known by the PT organiser.

To avoid collusion between the participants, test samples with three different layouts will be prepared and assigned randomly to the participating laboratories in each PT round.

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. Quality control is performed on behalf of the ENFSI Expert Working Group Firearms/GSR (as subcontractor) by an DIN EN ISO/IEC 17025:2018 [2] accredited laboratory. If, in the opinion of QuoData GmbH, any material does not meet homogeneity requirements, a replacement material is dispatched. Details on the performed tests, the results and the acceptability criteria are given in the final report of results of the respective PT round.

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 any problems with the condition of the test materials or accompanying documents are observed.

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 interest measuring 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 during the sample preparation process). The PbSbBa particles have to be searched and documented.

As the test sample nearly free from contamination, it is suitable for quick system validation checks and

quality assurance procedures. For protective reasons, the chip is coated with a thin carbon layer, which should help avoid charging. Nevertheless, if charging does occur, it is recommended to perform a supplementary carbon coating of the sample.

3.5 Metrological traceability

Metrological traceability is not possible because the appearance of the particles depends on the individual setting of the BSE value.

4 Analysis and reporting of results by the participants

4.1 Methods of analysis

Participants are asked to treat the PT sample in the same way as a routine sample. The analysis of the sample should be performed – where possible – with the same SEM/EDS parameter settings as used for routine casework with an 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 PbSbBa control pad is displayed in the lower left corner of the SEM screen (see example in 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 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 (BaF_2 was used for sample preparation). 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 to set the F-signal to zero. Any of these necessary changes of the standard settings should be reported as a comment in the answering form. Particles containing at least all three elements typical of GSR particles (Pb, Sb, and Ba) are to be classified as "characteristic" particles. Particles containing two of these three elements, i.e., particles containing PbSb, PbBa, or SbBa, should be reported as "consistent" particles. All other particles belong to the class "other" by definition of the particle classification scheme.

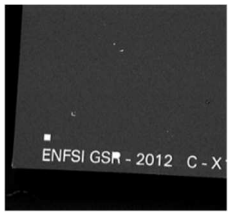

Figure 1: BSE image of the lower left corner part of a GSR PT test sample, here, a sample used in the GSR 2012 PT round

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 portal (https://forensics-pt.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).

4.4 Reporting format

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

- the absolute x and y co-ordinates (in µm or mm),
- beam x and y co-ordinates (only if a EDAX based FEI-GSR system is used)
- the corresponding particle sizes (calculated diameter in µm) and
- the unambiguous particle classification (3 classes: characteristic, consistent, and other – or corresponding unambiguous classes)

Results received after the deadline for any particular PT 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 GSR PT scheme coordinator.

No changes can be made if the cross-check period has expired. Results should be reported clearly, in the format requested. Incorrectly entered results will not be edited by QuoData GmbH.

4.5 Late return of results

Participants are asked to return their results before the 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 the 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 fifteen 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 example in Figure 2). Such a plot displays all correctly detected and classified 'regular' PbSbBa-particles in an XY-plot and enables the laboratory to check their submitted results.

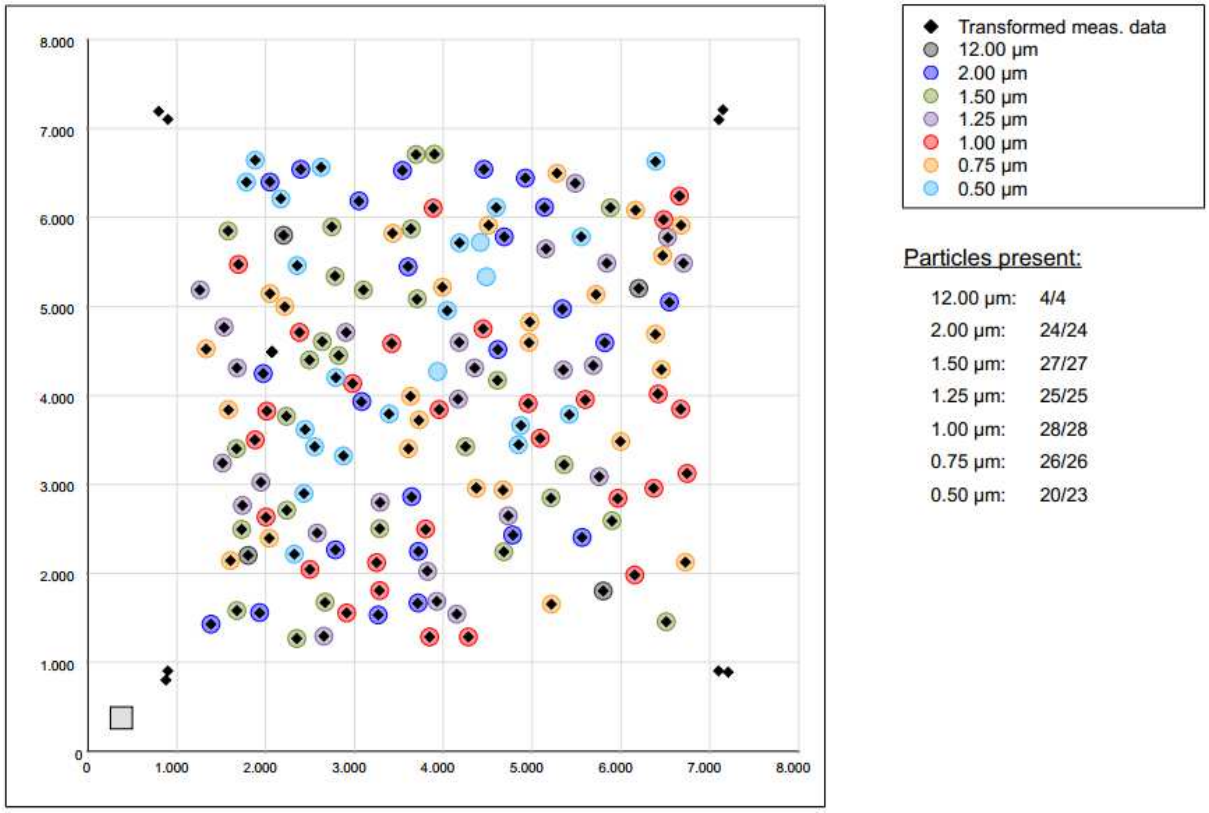

Figure 2: Individual result plot

5.2 Cross-checking

After the individual result plots have been made available to the laboratories in the GSR PT 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://forensics-pt.quodata.de

5.3 Statistical analysis

5.3.1 Assessment of the laboratories' performance

5.3.1.1 Assessment on the basis of the detection capability

For each laboratory, the 90 % detection capability, i.e. the particle size at which a 90 % probability of successful detection and classification is achieved by the laboratory, 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 three-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 13528:2022 [6] and EURACHEM [7]. The z score compares the difference between the participant's result x_i and the assigned value x_{pt} in terms of the acceptable spread of results or standard deviation for proficiency assessment σ_{pt}:

$$z = -\frac{x_i - x_{pt}}{\sigma_{pt}}$$

The assigned value x_{pt} is the consensus value of the laboratory-specific 90 % detection capability values 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 $u(x_{pt})$ and the reproducibility standard deviation σ_R across all laboratory-specific 90 % detection capability values by the following calculation:

$$\sigma_{pt} = \sqrt{u(x_{pt})^2 + \sigma_R^2}.$$

The reproducibility standard deviation[3] σ_R is calculated by the robust statistical Q method ([6], [8], [9]) according to ISO 13528:2022 [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].

[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.

5.3.1.2 Assessment on the basis of the number of correctly detected and classified PbSbBa particles

For information purposes only, the individual performance is also assessed on the basis of the number of correctly detected and classified PbSbBa particles. For each particle size evaluated and each laboratory, a z score is calculated. As assigned value x_{pt}, 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 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 Average performance

The average performance, i.e. the performance of a randomly selected laboratory [15], is displayed by the overall detection capability curve. In order to quantify the overall detection capability, the three-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 for the comparison of the method's detection capability of the current PT round to the method's detection capabilities obtained in former PT rounds.

5.3.3 Additional analyses

5.3.3.1 Comparison of laboratory performance between two PT rounds

Additionally, for those laboratories that participated in this PT scheme frequently, the running GSR PT scheme allows for a comparison of the results obtained in different PT rounds. Therefore, the z scores obtained in two subsequent PT rounds (the current PT and the previous PT round) are shown for the laboratories by means of a Youden plot [11]. Indeed, thanks to the standardization procedure involved in their calculation, z scores obtained by the same laboratory in different rounds can be meaningfully compared with each other, regardless of any differences between samples from one PT round to another. Hence, 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 laboratory detection capability curve and the obtained z score for the 90 % detection capability are provided with the final report of results.

z scores for the the number of correctly detected and classified PbSbBa particles are not provided in the indivudual certificate, but – for informative purposes only – in 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 four weeks after publishing the final report by contacting GSR-ENFSI-PT@quodata.de.

6 References and sources of information

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[6] ISO 13528:2022: *'Statistical methods for use in proficiency testing by interlaboratory comparison'*.

[7] EURACHEM Guide on "Selection, Use and Interpretation of Proficiency Testing (PT) Schemes by Laboratories"; Second Edition 2011; http://www.eurachem.org/images/stories/Guides/pdf/Eurachem_PT_Guide_2011.pdf.

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[15] Uhlig S., Niewöhner L., Gowik P.; *'Can the usual validation standard series for quantitative methods, ISO 5725, be also applied for qualitative methods?'*; Accreditation and Quality Assurance 16(11):533-537; 2011.