GENERAL PROTOCOL

PROFICIENCY TESTING SCHEMES

LGC Proficiency Testing
1 Chamberhall Business Park
Chamberhall Green
Bury, BL9 0AP
UK

Telephone: +44 (0) 161 762 2500
Email: axiopt@lgcgroup.com
Website: www.lgcstandards.com

Issue: 14 Issue date: November 2021

Record of issue status and modifications

ISSUE	ISSUE DATE	DETAILS	AUTHORISED BY
3	01/09/08	Updated with UKAS logo for single scope (0001) and removed reference to scheme year. Amended performance scoring to be consistent with requirements of ISO/IEC 17043.	T.Noblett
4	March 2010	Added generic details removed from individual Scheme Descriptions. Updated details following development of PORTAL. Included information about use of z' scores and rounding. Additional information added on assigned values and performance scores.	B.Brookman
5	November 2011	Updated address and accreditation details. Included information regarding scoring system for non-numerical assigned values. Added details for clinical schemes.	T.Noblett
6	April 2012	Updated references to annexes, titles and formatting. Updated details regarding number of results permitted for clinical PT schemes.	A.Fox
7	Sept 2014	4.2 Choice of Methodology Updated 5.4 Setting assigned values updated Annex II updated with details of when SMAD is used when MAD_E is 0 whilst calculating the Robust Standard Deviation.	A.Lane
8	November 2016	**Changes made throughout the document to reference ISO 13528 (2015)**5.5 & ANNEX IV Updates made to symbols to reflected the changes in ISO 13528 (2015)	Angela Lane
9	October 2017	Annex III amended to include note regarding measurements where replicates cannot be performed. Also updated regarding homogeneity procedure (and symbols) described in 13528 (2015).	T. Noblett
10	June 2018	Changes made through the document in regard to the PT Webshop. 2.8 Confidentiality details updated. 6.2 Renewal information updated. References and Sources of Information list updated.	K. Baryla
11	June 2019	5.5 Included information regarding calculation of the performance scores. Added an equation for calculating the expanded SDPA. ANNEX V updated.	K. Baryla
12	June 2020	LGC Standards Proficiency Testing replaced with LGC Proficiency Testing.	K. Baryla
13	Oct 2020	Removed Fax number, amended logo	A McCarthy
14	Nov 2021	4.4 and page 1 Updated email address 4.4 Added (15 for CLS participants) 5.1, 5.2, 5.7 Reference to semi-quantitative removed 6.3 Reference to fax removed Various typographical changes.	L. Eden

Notes:

Where this document has been translated, the English version shall remain the definitive version

INDEX

1 INTRODUCTION 4

1.1 Purpose and scope of proficiency testing 4
1.2 Quality Standards 4

2 SCHEME ORGANISATION 4

2.1 Scheme coordination and responsibilities 4
2.2 Use of Advisors and Advisory Groups 4
2.3 Management Committees 5
2.4 Typical scheme framework 5
2.5 Joining a PT scheme 5
2.6 Frequency of participation 6
2.7 Costs of participation 6
2.8 Confidentiality 6
2.9 Trials and new products 6

3 TEST MATERIALS 6

3.1 Test material preparation 6
3.2 Quality Control 7
3.3 Non-conforming products 7
3.4 Packaging and transportation 7

4 REPORTING OF RESULTS 7

4.1 Timescales 7
4.2 Choice of methodology 8
4.3 Reporting your results 8
4.4 Number of permitted results 8
4.5 Performance score calculator 9
4.6 Collusion and falsification of results 9

5 DATA ANALYSIS AND PERFORMANCE ASSESSMENT 9

5.1 Approaches to data analysis 9
5.2 Qualitative schemes 9
5.3 Quantitative schemes 9
5.4 Setting assigned values 10
5.5 Calculating z scores 10
5.6 Standard deviation for proficiency assessment (SDPA) 10
5.7 Interpreting results 10
5.8 Trend analysis 11

6 INFORMATION DISTRIBUTED TO PARTICIPANTS 11

6.1 Reports 11
6.2 Renewal information 11
6.3 Advice and feedback 11

ANNEX I - Scheme Operation Flowchart 13
ANNEX II - Procedure for calculating robust statistics 14
ANNEX III - General procedure and assessment criteria for a homogeneity check 15
ANNEX IV - Estimated Standard Uncertainty of the assigned value 16
ANNEX V - References and Sources of Information 17

1 INTRODUCTION

1.1 Purpose and scope of proficiency testing

Proficiency Testing (PT) is defined as the evaluation of participant performance against pre-established criteria by means of interlaboratory comparisons. The term 'External Quality Assessment' (EQA) may also be used to describe proficiency testing schemes within the clinical/medical sector.

LGC Proficiency Testing provides a wide range of schemes designed to facilitate the improvement of the quality of measurements in those sectors covered. Participation provides laboratories with a means of assessing the accuracy of their results and comparability to peer laboratories over time, and also provides information on technical issues and methodologies.

When performed within the context of a comprehensive quality assurance programme, proficiency testing is an independent means of assuring the quality of test and calibration results, as described in ISO/IEC 17025[1] and ISO 15189[2].

1.2 Quality Standards

International standards relevant to proficiency testing include ISO/IEC 17043[3] (2010) 'Conformity assessment – General requirements for proficiency testing' and ISO 13528[4] (2015) 'Statistical methods for use in proficiency testing for interlaboratory comparison'.

LGC Proficiency Testing is committed to continual improvement in quality and further information regarding our certification and accreditation to international quality standards is available on the LGC Proficiency Testing website www.lgcstandards.com.

Accreditation details for specific PT schemes can be found on the Scheme Application Forms and Scheme Descriptions.

2 SCHEME ORGANISATION

2.1 Scheme coordination and responsibilities

The day-to-day operation of each scheme is the responsibility of LGC Proficiency Testing. Individual schemes are managed by LGC Proficiency Testing Scheme Coordinators, responsible for customer service, technical and reporting functions. For some schemes, external advisors are used to provide the full range of relevant knowledge and expertise needed to operate the scheme effectively.

2.2 Use of Advisors and Advisory Groups

Technical expertise may be available in-house or may be provided by Advisors, either individually or as part of an Advisory Group. Advisors are selected on the basis of their technical knowledge and experience of the industry to which the scheme is related. Advisors may be used on an ad-hoc basis, being contacted when specific issues need to be addressed, or alternatively, formal advisory groups may be used. Advisory Groups consist of members who may or may not be participants on the scheme but who are experienced in the field of testing covered by the scheme.

The composition and terms of reference of each Advisory Group will be agreed on a scheme-by-scheme basis. Membership of the Advisory Groups is subject to change, but members' names are available on request.

For Advisory Groups, the Secretariat function will be provided by the Technical Scheme Coordinator. A member of LGC Proficiency Testing Management Team will also attend Advisory Group meetings, and chair where appropriate.

Advisory Groups will meet on a regular basis, usually at least once a year, to review the progress and performance of the scheme, and to provide advice on future operation and development of the scheme. A written record, in the form of minutes, will be kept of all Advisory Group meetings.

For some schemes the Advisory Group members (or an alternative group of experts) are responsible for providing expert opinions and interpretations against which the performance of the participants are assessed.

2.3 Management Committees

For schemes that are operated jointly with a partner organisation, a Management Committee may be set up to address business and operational issues for the scheme. The Management Committee, where constituted, will meet on at least an annual basis; a written record, in the form of minutes, will be kept of meetings. At least one member of LGC Proficiency Testing Management Team will be a member of each Management Committee.

2.4 Typical scheme framework

The structure within each scheme round is as follows:

- Participant orders processed and confirmed.
- Procurement, preparation, dispensing and quality control testing of test materials.
- Despatch of test materials to participants.
- Participants analyse the test materials and report their results to LGC Proficiency Testing as instructed, and within the specified deadline.
- Results analysed and the performance of laboratories assessed using appropriate statistical techniques.
- Reports written and issued to participants.
- Round reviewed and requirements for subsequent rounds identified.
- Commencement of next round.

Reports are issued as soon as possible after the round closure, although the timescale between closing dates and issue of the final report will vary from scheme to scheme. A flow diagram showing the typical process for a PT round is given in Annex I.

2.5 Joining a PT scheme

Application Forms are available for each scheme, and these include information about the distribution dates, the format and availability of test materials, and costs of participation. A Scheme Description is also available for each scheme, which provides technical and statistical information specific to that scheme.

In order to join a scheme, participants should complete the relevant Application Form, indicating which test materials they wish to receive during the scheme year. Alternatively, the participants can register for an account and place an order via our PT Webshop. If the availability of test materials changes during the scheme year, participants are kept fully informed. Most schemes do not have any restrictions to participation, but where these do occur this will be made clear on the Application Forms or through other documentation.

Once a completed Application Form or an order placed on PT Webshop is received, an Order Confirmation will be sent to the participant, confirming the test materials selected and distribution dates. Participants can amend an order up to one week prior to the distribution date, subject to test material availability. Any amendments to a participant's order will be confirmed to them in writing.

Participants are advised to participate in the scheme(s) that are most appropriate to their own area of testing. Where necessary, staff at LGC Proficiency Testing can advise on which scheme(s) are most suitable for participants.

2.6 Frequency of participation

Certain schemes have a minimum level of participation, whilst others have completely flexible participation. Third parties, such as retail groups, regulatory bodies and accreditation bodies may recommend minimum levels of participation. Details on frequency and participation will be provided on the scheme Application Forms and Scheme Descriptions.

2.7 Costs of participation

Costs for participation are reviewed annually and the current prices for each scheme are detailed on the scheme Application Form and on the PT Webshop. Payment terms are detailed in LGC Proficiency Testing' standard terms and conditions and on invoices. Non-payment or late payment may result in test materials and/or reports not being distributed.

2.8 Confidentiality

In order to ensure confidentiality, participants in all schemes are allocated a unique laboratory reference number. This number enables results to be reported without divulging the identities of participant laboratories. In cases where anonymity may have been breached, laboratory reference numbers may be changed on request from the participating laboratory, at the discretion of LGC Proficiency Testing. For some schemes, participants may agree to have their identity made known to others, but this will only be done with the knowledge and full permission of the participant. For clinical schemes, it is mandatory for EQA providers to provide reports on performance of UK participants who are involved in clinical care to the National Quality Assurance Advisory Panels; participants will be informed in the Application Forms, or in the PT Webshop, for the schemes to which this applies. In situations, when a regulatory authority requires proficiency testing results to be directly provided to the authority by LGC Proficiency Testing, the affected participants will be notified of this action in writing.

2.9 Trials and new products

LGC Proficiency Testing is continually striving to improve current schemes and to introduce new schemes/test materials/test parameters where appropriate. Before formally including in a scheme, new products may be introduced initially on a trial basis. It will be made clear to participants when they are participating in a trial.

3 TEST MATERIALS

3.1 Test material preparation

Test materials may come from a number of sources, and are carefully selected to meet the needs of participants. Wherever practical, test materials will be as similar as possible to those samples 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 or concentrated spiking solutions. The range of test materials will usually be

varied from round to round in order to be realistic and challenging. Details of individual test materials are available in the Scheme Description for each scheme.

3.2 Quality Control

A number of factors will be taken into consideration when determining the quality control testing required to be performed on each type of test material. These include, the degree of natural homogeneity, the stability of the test material, and the use of process control during production. Where undertaken, homogeneity assessment is carried out based on a procedure described in ISO 13528[4] (2015) 'Statistical methods for use in proficiency testing for interlaboratory comparison'. A full description of the procedure is included in Annex III. Further details regarding homogeneity testing are included in the Scheme Descriptions and/or reports.

For some schemes, for certain circumstances, homogeneity may not be undertaken on every test material type prior to despatch. This may be for operational reasons, or where the process has been proven to provide homogeneous samples. In these instances the participants' results are used to assess sample homogeneity and any issues will be treated as described below for non-conforming products.

3.3 Non-conforming products

Where, prior to dispatch, the homogeneity and/or the stability of test materials are not acceptable, the test materials will be withdrawn prior to distribution to participants. Where this may cause a delay in the distribution of test materials, participants will be informed. Occasionally, issues with test materials may not be identified until after distribution. Under these circumstances, this is taken into account when assessing participant results. The outcome will vary depending upon the situation but may involve; reporting of performance scores for information only, or the provision of replacement test materials. In these instances, full details will be provided to participants.

3.4 Packaging and transportation

Test materials are sent in appropriate packaging and under conditions intended to maintain the integrity of the test materials during transit.

Once packages have been delivered, LGC Proficiency Testing cannot be held responsible if they subsequently fail to reach the correct personnel or are not stored under the recommended conditions.

Participants are asked to check the contents of packages immediately on receipt and to contact LGC Proficiency Testing if there are any problems with the condition of the test materials or accompanying documentation. If packages are received damaged, then it would be very useful if participants could supply photographic evidence to assist our investigations.

4 REPORTING OF RESULTS

4.1 Timescales

To enable reports to be processed and issued as soon as possible after the closure of the proficiency test round, deadlines for the return of results are specified and must be adhered to. For certain test parameters there may be a date(s) specified by which examination of the test material is recommended to have been commenced and/or completed.
Results received after the reporting deadline cannot be included in the report. The main report is available to all participants subscribing to the round regardless of whether their results were submitted or not.

4.2 Choice of methodology

Participants are expected to use a technically appropriate test or measurement procedure of their choice, unless otherwise instructed. Participants are asked to treat the test material as a routine sample as much as possible.

When reporting results, participants are asked to select the best description of their method from a drop-down list of methods on the PORTAL reporting system. Only the most commonly reported methods will be included in the list, including standard or reference methods. Participants are asked to select the method which most closely describes their own method in use. If none of the methods listed are suitable, then 'Other' can be selected and a brief description of the method used recorded in the comments field.

This information is then used to produce a statistical summary of the most commonly reported methods for each analyte. These method summaries are given in the Scheme reports and enable the relative performance of each method to be compared.

4.3 Reporting your results

For the majority of schemes, results are returned through our bespoke electronic reporting software, PORTAL, full instructions for which are provided. For some schemes (or parts of a scheme) alternative reporting mechanisms are provided, details of which will be emailed to participants prior to test material receipt.

It is recommended that results and calculations are checked thoroughly before reporting. Results should be reported clearly, in the format and units detailed in the Scheme Description. If calculations are used, unless instructed otherwise, the laboratory is to report only the final calculated result. Part of the challenge of proficiency testing is the ability to perform calculations and transcribe results correctly. LGC Proficiency Testing staff cannot interpret or calculate results on participants' behalf. Once submitted and received, results cannot be amended and no changes can be made after the report has been issued.

In general, results of zero should not be reported; results should be reported depending upon the detection limit of the method used, for example, <10. Results of zero and truncated results, such as < or > cannot be included in the data analysis and therefore cannot be allocated a numerical performance score. The exception is a small number of parameters, where it may be appropriate to report a result of zero, depending on the measurement scale being used.

Results may be rounded up or down for the purposes of reporting and may not therefore be identical to the participant's original reported result. The effects of rounding may also mean that occasionally percentage totals do not add up exactly to 100%.

4.4 Number of permitted results

Although it is desirable for participants to submit multiple results in order to compare results between different analysts, methods or instruments, a single laboratory reporting a large number of results could potentially bias the dataset. In order to minimise the effects of bias, LGC Proficiency Testing limits the number of results participants are able to report. Each participant is able to enter up to 13 different results (15 for CLS participants). Of these results a maximum of 3 results can be 'nominated' results. Nominated results are included in the statistical analysis of the dataset, whilst non-nominated results are not. Nominated results must be obtained using different methods, again to minimise the effects of bias.
Further information is available in the PORTAL User Guide and the PORTAL Nominated Results FAQ, both of these documents are available for download from the PORTAL website and further information is available from axiopt.results@lgcgroup.com

4.5 Performance score calculator

For those schemes using a z/z' performance score, there is a performance score calculator available on the PORTAL website for those participants who missed the reporting deadline and wish to calculate their own performance scores.

4.6 Collusion and falsification of results

It defeats the objective of taking part in proficiency testing if participants are not returning genuine results. Certain measures are built into the scheme to try to prevent collusion, for example, assigned values are not made known to anyone before the report is issued and no results are accepted after the publication of the report. Participants will be contacted if there is clear evidence of collusion. However, ultimately the responsibility rests with each participant to behave in a professional manner.

5 DATA ANALYSIS AND PERFORMANCE ASSESSMENT

5.1 Approaches to data analysis

LGC Proficiency Testing organise a wide range of schemes, which may include qualitative, quantitative and interpretive measurements or tests. Different approaches to data analysis may therefore be used, the most common approaches being described below. Further information on the statistical approach for specific schemes is also provided in the Scheme Descriptions and Scheme Reports.

The advantages of using a performance score are:

- Results can be expressed in a form that is relatively easy to interpret and understand
- Results can be summarised in graphical or tabular form to depict overall performance
- A performance score allows participants to directly compare their own result with others
- If consistent statistical values are applied, a performance score enables participants to monitor trends in their own performance, over time.

When reviewing results, participants should take into account the methods used to analyse the data and to assess performance, and should review their performance in context, taking into account performance of the whole dataset.

5.2 Qualitative schemes

For qualitative tests, participant results will be compared against the intended result, also called the assigned value, based on formulation or expert assessment. A result which is the same as the assigned value is considered satisfactory. This approach is also used for quantitative tests when the target analyte is absent. For interpretive schemes where the result is subjective rather than quantifiable, a model answer produced by appropriate experts will be published in the report. In some qualitative and interpretative schemes a numerical score may be provided based on the expert judgement.

5.3 Quantitative schemes

For quantitative data, participants are assessed on the difference between their result and the assigned value (see 5.4); with this difference being represented by a performance score called a z or z' (z prime) score (see also Annex IV).

5.4 Setting assigned values

The assigned value is the value selected as being the best estimate of the 'true value' for the parameter under test. The method used to determine the assigned value may vary depending upon the particular scheme and test parameter, and is detailed in the relevant scheme description, along with details of the traceability in each case.

For quantitative tests, all assigned values are derived in accordance with ISO 13528. Where it is appropriate, practicable and technically feasible the assigned value will be derived through formulation (or occasionally through the use of a certified reference material) to provide metrological traceability; the associated uncertainty of the value can therefore be estimated. However, in most cases it will not be possible to use formulation or certified reference materials to set the assigned value and a consensus value will be the only practicable and technically feasible approach to use. When the assigned value is determined from the consensus value of participant results, or from expert laboratories, robust statistical methods are used for calculation of the consensus value, details of which are given in Annex II. The uncertainty of the assigned value is then estimated as described in Annex IV.

5.5 Calculating z scores

$$\text{z score} = \frac{(x_i - x_{pt})}{\sigma_{pt}}$$

where; $\underline{x_i}$ = the result reported by the participant
$\underline{x_{pt}}$ = the assigned value
σ_{pt} = standard deviation for proficiency assessment

The z score expresses performance in relation to an acceptable variation of the participant result to the assigned value. A z score of 2 represents a result that is 2 x σ_{pt} from the assigned value.

Where alternative scoring methods are used, full details will be given in the Scheme Description and/or report.

The returned results are rounded to the required number of decimal places specified in the Scheme Descriptions. The statistical calculations are performed on unrounded data and displayed as rounded to the required number of decimal places in the report.

5.6 Standard deviation for proficiency assessment (SDPA)

The method used to determine the SDPA may vary depending upon the particular scheme and test parameter. All SDPAs are derived in accordance with ISO 13528. When the SDPA is determined from the dispersion of participant results, robust statistical methods are used for the standard deviation, details of which are given in Annex II. A fixed, fit for purpose SDPA value is preferable as this enables performance scores to be compared from round to round to demonstrate general trends. This fixed value may be absolute or expressed as a percentage of the assigned value.

Where applicable, the value of SDPA is reported in the Scheme Description and/or report.

5.7 Interpreting results

For qualitative results, laboratories reporting the assigned result or range of results will be considered correct, and therefore have satisfactory performance.

For quantitative examinations, the following interpretation is given to z score results.

$\|z\| \leq 2.00$	Satisfactory result
$2.00 < \|z\| < 3.00$	Questionable result
$\|z\| \geq 3.00$	Unsatisfactory result

Where other performance techniques are used these are described in the Scheme Description and/or report.

For small data sets (generally with less than 8 results) there will be increased uncertainty around the assigned value if using consensus values from participants' results. For those analytes that use a formulation or reference value as the assigned value and a fixed fit for purpose SDPA (see 5.6) performance scores will be provided. Where the assigned value and/or SDPA is based on participant results, performance scores will be given for information only. For data sets with very limited results or where the spread of results is large, performance scores may not be provided. See also Annex IV with regards to where a z' score may be provided rather than a z score.

5.8 Trend analysis

A single result simply reflects the performance of the laboratory on the particular day that the test or measurement was carried out and can therefore only give limited information. Frequent participation in PT schemes over time can give greater insight into long-term performance and can help identify where an internal bias may be occurring. One of the best methods of summarising performance scores over time is graphically, as this gives a clear overview, and is less prone to misinterpretation than numerical methods. Participants are therefore advised to monitor their PT results over time. Further information regarding interpretation and trend analysis of proficiency results is given in the IUPAC 'International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories'[6], the Eurachem PT Guide[7] and ISO 13528.

6 INFORMATION DISTRIBUTED TO PARTICIPANTS

6.1 Reports

Reports are made available electronically. The contents of reports vary from scheme to scheme but include details of the composition of test materials, the assigned values, and tabular and/or graphical representations of participants' results and performance. Copyright to all reports remains with LGC Proficiency Testing but permission is granted to participants to make copies for their own internal use, for example for quality control and regulatory purposes. No other copies may be made without obtaining permission.

6.2 Renewal information

Renewal information will be sent to participants 2-3 months before the start of the new scheme year. The information sent will detail how to renew, including test material availability and changes since the previous scheme year. Relevant links to the PT Webshop will be provided or attachments in the form of the renewal letter, the Application Form, the Scheme Description and Terms and Conditions. Participants should review the new scheme year information and return their order to LGC Proficiency Testing, via the PT Webshop or using the Application Form either directly to the Bury (UK) office or through their local office.

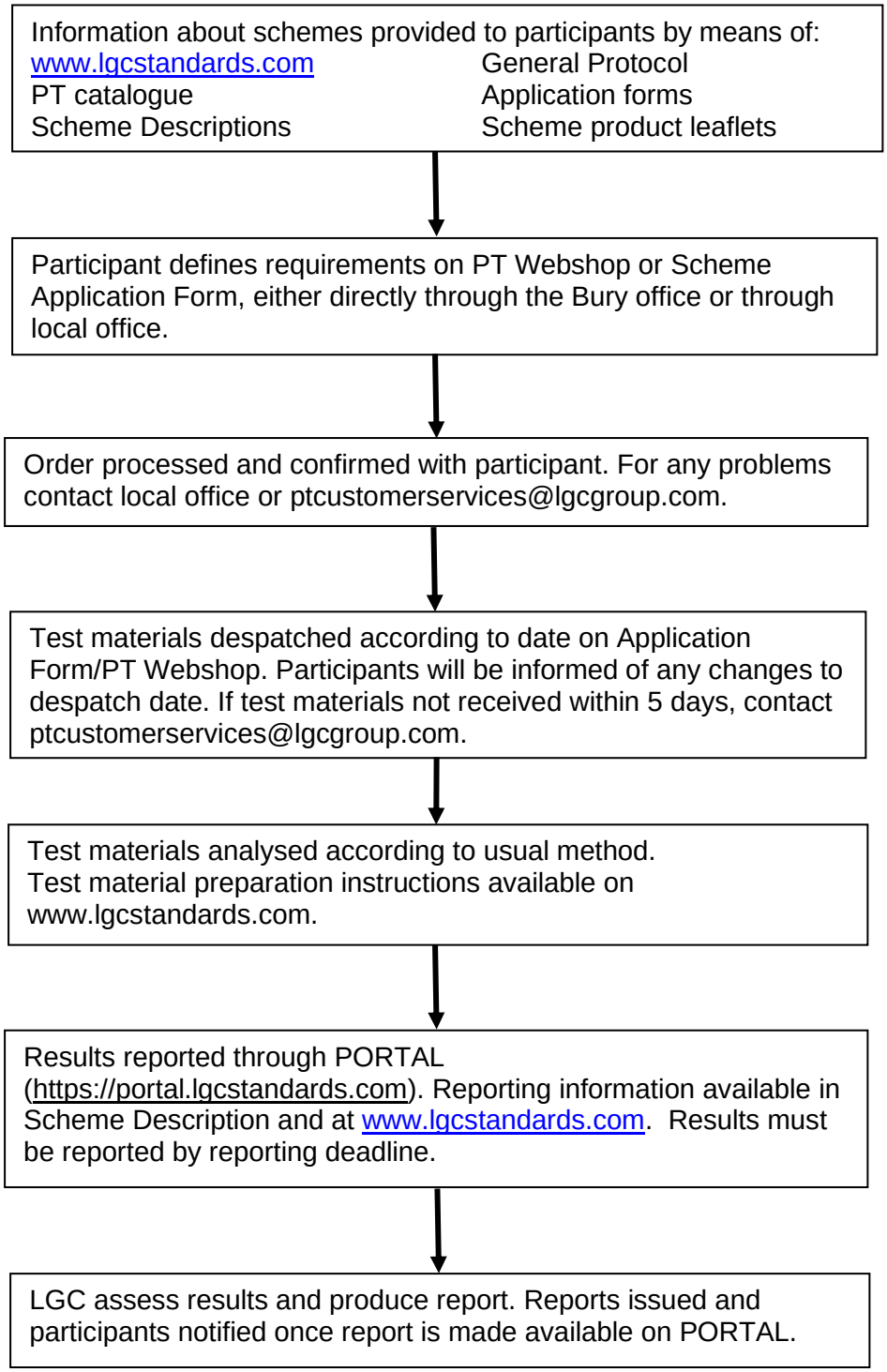
6.3 Advice and feedback

Communication with participants will be carried out through scheme-related documentation, e-mails, letters, newsletters or through distributors. Open meetings may also be organised and all interested parties invited to attend.

Part of the challenge of participating in a PT scheme is carrying out appropriate investigation and actions in response to an unsatisfactory or questionable result. Advice to participants who express concerns about their own individual performance is available through the Technical Scheme Coordinator. Additional test materials are usually available after each PT round to enable participants to repeat testing if necessary.

Comments on any aspect of the scheme are welcome either by e-mail, phone or letter. In the event 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 the results of any investigations carried out will be communicated, as appropriate, to the participant.

ANNEX I - Scheme Operation Flowchart



ANNEX II - Procedure for calculating robust statistics

Robust mean (median)

The consensus value can be calculated using the robust mean of all participant results. In LGC PT schemes the robust mean used is the median. If the data, where there are an odd number of results are arranged in order of magnitude ($x_1, x_2, \ldots\ldots, x_n$) the median is the central member of the series, i.e. there are equal numbers of observations smaller and greater than the median. Where there is an even number of results, the median is the average of the middle pair of numbers within the series. For a normal distribution the mean and median have the same value. The median is more robust, in that it is virtually unaffected by extreme values.

Robust Standard Deviation

In LGC PT schemes the normalised Median of Absolute Deviations (MAD_E) from the sample median is used as a robust standard deviation.

$MAD = \text{median}_{i = 1,2,\ldots\ldots n}(|x_i - X|)$ where n = number of results

For example:

Data (g)	5·6	5·4	5·5	5·4	5·6	5·3	5·2
Ordered Data	5·2	5·3	5·4	**5·4**	5·5	5·6	5·6

Sample median = 5.4

$\lvert x_i - X \rvert$	0·2	0·1	0·0	0·0	0·1	0·2	0·2
Ordered Difference	0·0	0·0	0·1	**0·1**	0·2	0·2	0·2

Therefore MAD = 0.1
MAD is then scaled by a factor of 1.483 to make it equivalent to a normal deviation (MAD_E).
Hence $MAD_E = 1.483 \times MAD = 0.1483$

If MAD_E is equal to zero SMAD should be calculated:

$SMAD = \text{mean}_{i = 1,2,\ldots\ldots n}(|x_i - X|) \times 1.2531$

The Robust Standard Deviation may be used as the Standard Deviation for Proficiency Assessment (SDPA) for calculation of z-scores. Other statistical methods for the calculation of robust estimators are available.

Removal of errors and blunders

Although robust estimators are used in order to minimise the influence of outlying results, extreme results or results that are identifiably invalid should not be included in the statistical analysis of the data. For example, these may be results caused by calculation errors or the use of incorrect units. However, such results can be difficult to identify by the PT organiser. For this reason, the robust mean and standard deviation will be calculated as above, but those results that are out of the range of the assigned value ± 5 x SDPA will be excluded and the robust mean and standard deviation will then be recalculated. These recalculated values will be used for the statistical analysis. All results, including excluded results, will be given performance scores.

ANNEX III - General procedure and assessment criteria for a homogeneity check

Test materials are assessed for homogeneity using procedures described in Annex B of ISO 13528[4] (2015). A brief description of the procedure is described below;

a) Choose a property (or properties) to be assessed for homogeneity.
b) Choose a laboratory to carry out the homogeneity check and the measurement method to use. The method should have a sufficiently small repeatability standard deviation (s_r) so that any significant inhomogeneity can be detected. If possible, s_r should be less than 0.5 x σ_{pt} *(*the standard deviation for proficiency assessment).
c) Prepare and package the proficiency test items for a round of the scheme ensuring there are sufficient items for the participants and the homogeneity check.
d) Select a number *g* of the proficiency test items in their final packaged form using a suitable random selection process, where g $\geq$ 10. This number may be reduced if suitable data are available from previous homogeneity checks on similar proficiency test items prepared by the same procedures.
e) Prepare *m* $\geq$2 test portions from each proficiency test item using techniques appropriate to the proficiency test item to minimise between-test-portion differences.
f) Taking the *g* x *m* test portions in a random order, obtain a measurement result on each, completing the whole series of measurements under repeatability conditions.
g) Calculate the general average *x,* within-sample standard deviation s_w, and between-sample standard deviation s_s.

NOTE When it is not possible to conduct replicate measurements, for example with destructive Tests, then the standard deviation of the results can be used as s_s.

h) Examine the results to look for possible trends in analysis or production order and to compare differences between replicates.
i) Compare the between-sample standard deviation s_s with the standard deviation for proficiency assessment σ_{pt}. The proficiency test items may be considered adequately homogenous if $s_s \leq 0.3\sigma_{pt}$.

NOTE When the above criterion is met then the between-sample standard deviation contributes less than 10% of the variance for evaluation of performance.

j) Calculate the allowable sampling variance $\sigma^2_{allow} = (0.3 \times \sigma_{pt})^2$
k) Calculate $c = F_1\sigma^2_{allow} + F_2 s^2_w$, where s_w is the within-sample standard deviation and F_1 and F_2 are from standard statistical tables as shown below:

m	20	19	18	17	16	15	14	13
***F*1**	1.59	1.60	1.62	1.64	1.67	1.69	1.72	1.75
***F*2**	0.57	0.59	0.62	0.64	0.68	0.71	0.75	0.80

m	12	11	10	9	8	7	6	5
***F*1**	1.79	1.83	1.88	1.94	2.01	2.10	2.21	2.37
***F*2**	0.86	0.93	1.01	1.11	1.25	1.43	1.69	2.10

If $s_s > \sqrt{c}$, then there is evidence that the batch of proficiency test items is not sufficiently homogenous.

ANNEX IV - Estimated Standard Uncertainty of the assigned value

The assigned value (x_{pt}) has a standard uncertainty ($u(x_{pt})$) that depends upon the method used to derive the assigned value. When the assigned value is determined by the consensus of participants' results, the estimated standard uncertainty of the assigned value can be calculated by;

$u(x_{pt})$ = 1.25 x Robust standard deviation/$\sqrt{n}$ where n = number of results

When the assigned value is determined by formulation, the standard uncertainty is estimated by the combination of uncertainties of all sources of error, such as gravimetric and volumetric measurements.

If $u(x_{pt})$ is ≤ 0.3 x SDPA, then the uncertainty of the assigned value can be considered negligible and need not be considered in the interpretation of results.

If $u(x_{pt})$ is > 0.3 x SDPA, then the uncertainty of the assigned value is not negligible in relation to the SDPA and so z' (z prime) scores, which include the uncertainty of the assigned value in their calculation, will be reported in place of z scores.

z' scores are calculated as follows:

$$z' = \frac{(x_i - x_{pt})}{\sqrt{\sigma_{pt}^2 + u(x_{pt})^2}}$$

Where
$\underline{x_{pt}}$ = the assigned value
$\underline{x_i}$ = participant result
σ_{pt} = standard deviation for proficiency assessment
$u(x_{pt})$ = standard uncertainty of the assigned value $\underline{x_{pt}}$

Expanded SDPA = $\sqrt{\sigma_{pt}^2 + u(x_{pt})^2}$

The magnitude of z' scores should be interpreted in the same way as z scores.

ANNEX V - References and Sources of Information

[1] ISO/IEC 17025 (2017) ‘General requirements for the competence of testing and calibration laboratories’.

[2] ISO 15189 (2012) ‘Medical laboratories - Requirements for quality and competence’.

[3] ISO/IEC 17043 (2010) ‘Conformity assessment – General requirements for proficiency testing’.

[4] ISO 13528 (2015) ‘Statistical methods for use in proficiency testing by interlaboratory comparison’.

[5] UKAS (United Kingdom Accreditation Service), 21-47 High Street, Feltham, Middlesex, TW13 4UN.

[6] M Thompson, S L R Ellison, R Wood, ‘International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories’, Pure Appl. Chem., 2006, 78, 145-196.

[7] B. Brookman and I. Mann (eds.) Eurachem Guide: Selection, Use and Interpretation of Proficiency Testing (PT) Schemes (3rd ed. 2021). Available from www.eurachem.org.