General Procedure and Criteria

IARMA Proficiency Tests

1 INTRODUCTION

1.1 Purpose and scope of proficiency testing

Proficiency testing (PT) is defined as the evaluation of laboratories performance against pre-established criteria by means of interlaboratory comparisons. Participation in PT has several objectives:

- to verify analytical performance, identify any shortcomings and their source and magnitude;
- to validate procedures and to demonstrate the reliability and validity of the measurement results;
- to compare laboratory performance with peers;
- to identify shortcomings which are not detected within the internal quality system;
- to meet the ISO 17025 requirements.

IARMA Proficiency Tests provide a tool to facilitate the improvement of the quality of measurement results to fit for applications in environmental radioactivity. Participation provides laboratories with a mean of assessing not only the trueness but also the precision and comparability of their results to peer laboratories over time.

Participation in proficiency tests should be considered within the context of a comprehensive quality assurance system as an independent mean of assuring the quality of test and calibration results, as described in ISO/IEC 17025 [1].

1.2 Relevant Standards

IARMA assures that the provided proficiency tests are conducted according to the requirements of the International standards relevant to proficiency testing namely: ISO Guide 35 [2] 'General requirements for the competence of reference materials producers', ISO/IEC 17043 [3]
'Conformity assessment – General requirements for proficiency testing' and ISO 13528 [4]
'Statistical methods for use in proficiency testing for interlaboratory comparisons'.

Internal audits and peer-review audits are planned and executed to assure that quality criteria relevant to PTs organization are met.

2 PROFICIENCY TEST ORGANISATION

2.1 Proficiency Test management and responsibilities

PT is conducted according to a planned project where the objective of the PT is defined and all steps of PT material preparation and certification are described. A time schedule for the implementation of the PT is announced and communicated to participants.
The everyday activities of each PT is the responsibility of IARMA Chief Technical Officer (CTO).
IARMA CTO is responsible for customer service, planning, technical preparation, coordination with Collaborating Institutions and reporting activities.

2.2 Use of Collaborating Institutions and Scientific Advisory Panel

During PT planning and implementation, regular consultations and reviews are provided by the specialists of Collaborating Institutions and Scientific Advisory Panel to assure the quality of the PTs and to assure that the PT objective is met.

Advisors may be used on an informal basis, being contacted when specific issues need to be addressed, or alternatively, formal Scientific Advisory Panel may be used.

The members and terms of reference of the Scientific Advisory Panel will be agreed on PT by-PT basis. Membership of the Scientific Advisory Panel is subject to change, but members' names are available on request. Members of the Scientific Advisory Panel are asked to sign a confidentiality undertake.

IARMA CTO takes the responsibility for the Secretariat function of the Scientific Advisory Panel and will attend its meetings.

The Scientific Advisory Panel will meet on a regular basis, usually at least once a year, to review the progress and performance of PTs, and to provide expert advice on future plans and development of future PTs. A written record, in the form of minutes, will be kept of all of the Scientific Advisory Panel meetings.

2.3 Proficiency test sequence of actions

The work sequence within each PT round is as follows:

- Preparation and approval of the project plan of the PT.
- On-line registration is open for participants.
- Participant registrations processed and confirmed.
- Procurement, preparation, spiking, homogeneity and stability testing of PT materials.
- PT material characterization campaign in expert laboratories.
- Assignment of target values and associated uncertainties.
- Dispatch of test materials to participants.
- Participants analyse the PT materials and report their results to IARMA via web portal.
- Participants results are evaluated and the performance of laboratories assessed using appropriate statistical methods.
- Send individual evaluation report within one week of results reporting closing date.
- Publish summary PT report within two months of closing date.
- Convene the annual technical workshop to discuss the PT results.
- Set up a project plan and requirements for the following PT identified.

2.4 Register for a PT

On-line application Forms are available for each PT, and this includes description of the PT materials, list of measurands, PT time schedule and PT fees.

In order to join a PT, participants should complete the relevant Application Form, indicating contacts details, shipment and billing addresses and which test materials they wish to receive. Upon receipt of the registration, an order confirmation will be sent to the participant, indicating the test materials ordered and expected delivery dates.

2.5 Costs of participation

Fees for participation are reviewed annually and the current fees for each PT are detailed on the registration application form. Payment terms are detailed on the registration page and on invoices sent to the participants.

2.6 Confidentiality

In order to meet confidentiality requirement, participants in all PTs are assigned a unique laboratory code which is changed every PT. This code enables performance evaluation to be published without divulging the identities of participant laboratories. The CTO is the only IARMA staff member who is responsible for assignment of the laboratory codes.

Information on the analytical performance of the participating laboratories could be passed to a third party only after receiving a written request from the concerned laboratory. Participating laboratory may request in writing to have its code made known to its customers.

Electronic information and internal records are kept in a safe and secure manner to assure information confidentiality and integrity.

3 TEST MATERIALS

3.1 Proficiency Test material preparation

IARMA proficiency test materials are mainly natural matrices to simulate the real analytical challenge in a laboratory and to make them as similar as possible to those samples routinely tested by participating laboratories.

To meet specific PT objectives and if natural matrices are not available, spiked PT materials are used. The IARMA spiked PT materials are prepared according to a validated procedure and are subject to a rigorous quality control procedure to assure homogeneity, stability and metrological traceability as described in [5].

IARMA PT materials are characterized by group of selected expert laboratories to assign the target values and associated uncertainties prior to distribution to participating laboratories.
The selection of expert laboratories is based on their demonstrated analytical performances through application of a quality assurance system including method validation and well established uncertainty budget and on good analytical performance in proficiency tests.

The range of the activity in the PT materials will usually be varied from run to run in order to be realistic and challenging. Details of annual PT materials are available in the PT announcement for each year.

3.2 Homogeneity study

The PT materials homogeneity study is necessary to demonstrate that the different bottles of the PT material produced are sufficiently homogeneous in terms of within bottle and between bottles. It is also important to demonstrate that the variations associated with the property values of interest are within the stated uncertainties and suitable for the intended use of the PT material. The between-bottle study allows the estimation of one of the uncertainty components considered in the combined uncertainty of the target value of the PT material. The homogeneity study will also assure that the production process of the PT material is under statistical control and does not suffer from any trend.

The homogeneity assessment is carried out based on the procedure described in the ISO Guide 35 [6] IUPAC International Harmonized Protocol for the proficiency testing of analytical chemistry laboratories [7].

Linear regression fitting is applied to the results and one-way analysis of variance (ANOVA) is used to check the significance of the regression coefficients [6]. The results are checked to verify if any trends occurred in the measurement order and/or the bottling of the material.
Single way ANOVA for is also applied to assess the between bottles variability.

The uncertainty component associated with between-bottles heterogeneity is calculated according to ISO Guide 35 using the ANOVA outputs and Equation 1.

$$u_{bb}^{*}=\sqrt{\frac{MS_{within}}{n}}\sqrt[4]{\frac{2}{v_{MS_{within}}}} \qquad (1)$$

Where:

n	represents the number of measurements per bottles;
$MS_{between}$	is the between bottles mean square of the ANOVA test;
MS_{within}	is the within bottle mean square of the ANOVA test;
vMS_{within}	is the within bottle degrees of freedom.

3.3 Stability study

Stability study is one of the steps in the PT materials production and it is composed of short and long-term stability studies [6]. The short-term stability study is related to the definition of transport conditions in which the material will not suffer degradation. The long-term stability study is used to verify if the new PT material may be stored at defined conditions such as room temperature.

The quality and stability of the IARMA PT materials are assured during the whole period of the PT run. Long term stability results will be communicated to customers and the estimated uncertainty associated with long term storage will be added to the uncertainty budget of the measurands.

3.4 Packaging and transportation

PT materials are sent in appropriate and validated packaging in a manner to maintain the integrity and stability of the test materials during transportation.
Upon receipt of the PT package, participants should check the inventory of the package and compare its contents with the items listed in the “Packaging list”. Participants are requested to sign and date the “Acknowledgment of receipt of PT materials”, and to send it to IARMA.

If the package content does not confirm to the purchase order or if any damage or leakage was observed IARMA should be informed as soon as possible.

In addition, participants should assure that PT package reaches the correct personnel and to be handled and stored under the recommended conditions.

4 REPORTING OF RESULTS

4.1 Measurement results and uncertainties

Within the announced time schedule of the PT, participants are asked to report measurement results and estimated standard combined uncertainties using the on-line Portal reporting application.

In order to assess the analytical performance and potential source of discrepancies, participants are requested to provide technical information on the analytical method used in the PT. For this purpose, a dedicated on-line simplified form is available. On the Portal reporting application, a drop menu of the most common methods will be available to select from.

Results should be reported after being checked, in the same units as requested in the on-line reporting application. Results are evaluated as it is reported without any interpretation or calculation. Results could be amended on-line as long as Portal system allows it and before divulgating thetarget values of the PT and no changes can be made after the individual evaluation report has been issued.

Reported measurement result uncertainty should be realistically estimated as its under or over estimation could affect the final performance score.

After reporting the results and uncertainties along with the technical information, participants are requested to send a signed print out from the system as a reference of the data entered. The print out will be used as the definitive source of information to confirm your results transferred to the data base and to accept any claim in case of data transfer errors.
The print out could be sent by email or fax.

Results received after the distribution of the individual evaluation reports cannot be included in the summary report. The summary report will be distributed to all participants registered to the PT regardless of whether their results were submitted or not.

4.2 Choice of methodology

Participants may use any routine method of their choice which is considered technically appropriate. Participants are asked to treat the test material in the same way as they would a routine sample.

Participants should homogenize the PT material before each sub-sampling and to let the powder of the solid PT material to settle down before opening the bottle. Participants are asked to take all necessary precautions when opening the sample bottle to prevent any spread of the material in the laboratory.

The stated minimum sample intake should observed and not to take smaller sample amount to assure that within bottle homogeneity is within the stated uncertainty range.

5 DATA ANALYSIS AND PERFORMANCE ASSESSMENT

5.1 Approaches to data analysis

Several performance indicators are calculated to allow participants to compare their performance in different PT schemes.

The general principles of IARMA approach in using of performance indicators are:

- Both bias and trueness of reported results are evaluated;
- Performance indicators values could be interpreted to make conclusion on the root cause of a shortcoming;
- Performance indicators are comparable between participants and different PT runs;
- Level of performance could be monitored and checked for trends;

5.2 Assignment of target values and uncertainties

The measurement results of the PT material characterization campaign are checked for compliance with the reporting requirements in terms of the requested technical information and details of the procedure.

The collected set of data from the group of expert laboratories of studied elements are subjected to several statistical tests. Beside general descriptive statistics, the following tests are performed at a 95% confidence level:

- Outlier tests (Dixon, Grubbs);
- Directional tests (Skewness, Kurtosis);
- Normality test (Kolmogorov-Smirnov-Lilliefors).
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If an outlier is detected, the result is discussed with the analyst, a result could be eliminated only based on technically justified reason. In addition, the directional tests and Kolmogorov-Smirnov-Lilliefors normality test are used to check the conformity to normal distributions of the data sets for all elements.

The target value of studied elements in the PT material are derived as a consensus of all accepted results. The median, arithmetic mean, Algorithm A mean and Hampel mean as described in ISO 13528 [4], are calculated and compared with the median. In most case, the median is adopted as an estimator of the target value, rounded off to the most significant number of the uncertainty. According to the ISO Guide 35 [6] the combined uncertainty associated with the target value consists of uncertainties related to characterization (uchar), between bottle heterogeneity (ubb) and long–term stability (ustab). These different uncertainty components are estimated and propagated to estimate the combined standard uncertainty of the reference value of studied elements (uCRM) as described in Equation 5.

$$u^2_{\text{target}} = u^2_{\text{char}} + u^2_{\text{bb}} + u^2_{\text{stab}} \tag{2}$$

The uncertainty component associated with the characterization (uchar) is estimated using the approach described in ISO 13528 [8]. In this approach, the uncertainty of characterization is estimated according to Equation 2.

$$u_{\text{char}} = \frac{STD}{\sqrt{n}} \tag{3}$$

where *STD* is the standard deviation of reported results in the characterization campaign and n is the number of reported results.

5.3 Performance indicators

The proficiency test results are evaluated against the acceptance criteria for trueness and precision and assigned the status “Acceptable”, “Warning” or “Not Acceptable” accordingly [8].

Assessment of Trueness

The participant result is assigned “Acceptable” status for trueness if:

$$A1 \leq A2 \tag{4}$$

where:

$$A1 = \left|\text{Value}_{\text{target}} - \text{Value}_{\text{reported}}\right| \tag{5}$$

$$A2 = 2.58 \times \sqrt{u^2_{\text{target}} + u^2_{\text{reported}}} \tag{6}$$

Assessment of the claimed uncertainty

To evaluate the claimed measurement result uncertainty an estimator P is calculated for each reported uncertainty, according to the following formula:

$$P = \sqrt{\left(\frac{u_{\text{target}}}{\text{Value}_{\text{target}}}\right)^2 + \left(\frac{u_{\text{reported}}}{\text{Value}_{\text{reported}}}\right)^2} \times 100\% \tag{7}$$

P directly depends on the measurement result uncertainty claimed by the participant. The Limit of Acceptable Precision (LAP) for each analyte respectively is defined for the respective proficiency test in advance, including any adjustment due to the concentration or activity level of the analytes concerned and the complexity of the analytical problem.

Participants’ results are scored as “acceptable” for the claimed uncertainty when P ≤ LAP.

Assessment of relative bias

To evaluate the bias of the reported results, the relative bias between the reported value and the target value is calculated and expressed in percentage:

$$Bias_{relative} = \frac{Value_{reported} - Value_{target}}{Value_{target}} \times 100\% \quad (8)$$

Assessment of z score

The z-score is calculated from the laboratory results, the target value and a standard deviation in accordance with the following equation:

$$z_{Score} = \frac{Value_{reported} - Value_{target}}{STD} \quad (9)$$

On the basis of the "fitness for purpose" principle, the target standard deviation (STD) is:

$$0.10 \times Value_{target} \quad (10)$$

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 STD from the assigned value.

5.7 Interpreting results

In the final evaluation, both scores for trueness and precision are combined. A result must obtain an "acceptable" score in both criteria to be assigned the final score "acceptable". Obviously, if a score of "not acceptable" was obtained for both trueness and precision, the final score will also be "not acceptable". In cases where either precision or trueness is "not acceptable", a further check is applied. The reported result relative bias (R. Bias) is compared with the maximum acceptable bias (MAB). If R. Bias ≤ MAB, the final score will be "acceptable with warning". "Warning" will reflect mainly two situations. The first situation will be a result with small measurement uncertainty; however its bias is still within MAB. The second situation will appear when result close to the assigned property value is reported, but the associated uncertainty is large. If R. Bias > MAB, the result will be "not acceptable".

For the relative bias the following interpretation is given.
$|Bias_{relative}| \leq 20\%$ Acceptable score
$20 < |Bias_{relative}| < 30$ Warning score
$Bias_{relative} \geq 20$ Not acceptable score.

For z score the following interpretation is given.
$|z| \leq 2.00$ Acceptable score
$2.00 < |z| < 3.00$ Warning score
$|z| \geq 3.00$ Not acceptable score.

6 FEEDBACK AND COMMUNICATION TO PARTICIPANTS

6.1 Individual Evaluation Reports

Five working days after the closing date for results reporting, an individual evaluation report is issued for each laboratory which can be downloaded from the Portal application. Reports are made available electronically, signed and stamped paper copies are also available.
The individual evaluation report includes details of the target values and associated uncertainties, evaluation criteria and laboratory performance scores.

6.2 Summary PT report

The summary PT report is issued within two months of the reporting closing date. It contains the description of PT material preparation, assignment of target values and uncertainties and tabular and/or graphical representations of participants' results and performance.

6.3 Annual workshop and feedback

A scientific annual workshop is organized and all interested parties invited to attend at no fees to excluding travel and accommodation fees.

One of the most important advantages of PT participation is to find out the root cause of a performance shortcoming and to take appropriate actions to remediate it. To overcome this technical challenge, professional advice could be provided to laboratories upon request.
Comments and/or suggestions on any aspect of the PT are welcome either by e-mail, phone, fax 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.
General Protocol

References

[1] ISO/IEC 17025 (2005) '*General requirements for the competence of testing and calibration laboratories'.*
[2] ISO Guide 34 (2009) '*General requirements for the competence of reference materials producers'.*
[3] ISO/IEC 17043 (2010) *'Conformity assessment – General requirements for proficiency testing'.*
[4] ISO 13528 (2005) *'Statistical methods for use in proficiency testing by inter-laboratory comparisons'.*
[5] A. Shakhashiro, A. Gondin da Fonseca Azeredo, U. Sansone, A. Fajgelj, '*Matrix materials for proficiency testing: optimization of a procedure for spiking soil with gamma-emitting radionuclides', Analytical and Bioanalytical Chemistry, 2007, 387(7), p. 2509-2515, doi 10.1007/s00216-006-0772-z.*

[6] ISO Guide 35 (2006), 'Reference materials – General and statistical principles for certification'.

[7] 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.

[8] A. SHAKHASHIRO, A. FAJGELJ, U. SANSONE, 'Comparison of Different Approaches To Evaluate Proficiency Test Data, Presented and accepted in the publications of the International Workshop on Combining and Reporting Analytical Results'. The Role of (metrological) Traceability and (measurement) Uncertainty for Comparing Analytical Results, Rome 6-8 March, 2006.