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Information on Proficiency Testing Schemes organized by the National Water Reference Laboratory (NRL) of the Water Research Institute (WRI)

Introduction

The objective of the proficiency testing scheme (PTS) is to ensure regular external control and to achieve comparability of the results among different laboratories.

Participation in PTS allows laboratories to demonstrate the quality of their performance. At the same time PTS serve to identify potential source of errors in the analytical procedures used by laboratories. Whit a properly functioning feedback in the quality system, the proficiency testing allows laboratories to improve the quality of their performance and the results of analyses.

Definitions/terms [1]

- Proficiency testing - evaluation of participant performance against preestablished criteria by means of interlaboratory comparisons
- Proficiency testing scheme (PTS) – proficiency testing designed and operated in one or more rounds for a specified area of testing, measurements, calibration or inspection
- Proficiency testing round – single complete sequence of distribution of proficiency test items, and the evaluation and reporting of results to the participants
- Interlaboratory comparison – organization, performance and evaluation of measurements or tests on the same or similar items by two or more laboratories in accordance with predetermined conditions
- Proficiency testing item – sample, product, artefact, reference material, piece of equipment, measurement standard, data set or other information used for proficiency testing
- Proficiency testing provider – organization which takes responsibility for all tasks in development and operation of a proficiency testing scheme
- Coordinator of PTS – one or more individuals with responsibility for organizing and managing all of the activities involved in the operation of a proficiency testing scheme
- Customer – organization or individual for which a proficiency testing scheme is provided through a contractual arrangement
- Participant – laboratory, organization or individual that received proficiency test items and submits results for review by proficiency testing provider
- Accreditation - official recognition that the workplace is competent to perform certain measurements/analyses or certain types of measurements/analyses.

Competence of the NRL as an provider of PTS

The Water Research Institute (WRI) has been organizing and coordinating inter-laboratory comparison tests for hydro-analytical laboratories in Slovakia since 1987. NRL was accredited for this activity in November 2006 and re-accredited in January 2010 in accordance with the requirements under the legal documents [1], [2]. Activity specification of the NRL as a provider of PTS is documented in the Annex to the Certificate including the following:

- The scope of proficiency testing,
- Properties – parameters being compared,
- Range of values being compared,
- Proficiency testing repetition interval.

Analysed parameters (compared properties) are included in the seven groups:

- Basic physical-chemical analysis for drinking/surface water (ZPV) and wastewater (ZOV);
- Trace inorganic analysis (SAA) and trace organic analysis (SOA), radio-chemical analysis (RR);
- Microbiological (MBR) and hydro-biological analysis (HBR), eco-toxicological tests (ETS),
- Sampling of drinking water, surface water and waste water (OPiV, OPoV, OOV).

Proficiency testing schemes are aimed at three types of tests:

- inter-laboratory comparison tests
- known-value schemes
- partial-process schemes

Organizing the Inter-laboratory Comparison Test (ICT)

PTS organized by NRL is related to the analysis of drinking water, surface water and waste water in accordance with the requirements under the Regulation of the Government SR 496/2010 laying down requirements for water intended for human consumption and quality monitoring of water intended for human consumption and the Regulation of the Government SR 269/2010 laying down requirements for achieving good status of water.

Samples for ICT are prepared and analyzed in accredited NRL testing laboratory [3].

Besides determining the required water quality parameters at sampling site and parameters in taken water samples, the sampling techniques and methods, sampling devices, related documentation and tests of theoretical knowledge are evaluated within the PTS focused on water sampling in accordance with the requirements under the current legislation. Samplings are carried out at selected sampling sites.

Participation in ICT organized by NRL

PTS are usually available without any restrictions. Any number of laboratories from different areas of interest can participate in PTS. The only restriction is that the requirements for related type of PTS must be met.

New applicant for participation in PTS applies for registration to the NRL Register by submitting completed Application Form for Registration to the NRL Register. For the confirmation of registration the applicant is provided with the copy of confirmed application form.

Participation in PTS is anonymous. Participants are registered in the NRL database under the permanent registration number that is used for regular contact / communication with the ITC participants. In each ICT project, the participants are assigned an identification code. It is a randomly generated number that varies for each ICT and is communicated only to a particular participant in ICT. The results are presented in the ICT final reports under this identification code.

Registration Form for Inter-laboratory Comparison Test

At the beginning of each year, the NRL prepares an annual plan of proficiency testing scheme, where the following information is provided:
- Field of activity (chemical, biological methods),
- Name and designation of each ICT,
- Dates of testing,
- Parameters to be monitored and matrices to be used.

Offer – Binding Order

The ICTs are scheduled according to the annual plan. Potential participants are provided with information on the planned ICT sufficiently in advance (usually two months). The offer for a particular ICT in a form of “binding application-order” is sent to laboratories that are included in the NRL Directory based on previous participation or laboratories which ask for inclusion in the PTS. The offer proposal contains detailed information about a specific ICT such as the date of ICT, prepared parameters, prices, deadline for submitting applications, method of distribution, distribution center, etc.

If necessary, the laboratory indicates in the application form its identity, required parameters, place of samples delivery and total price of the order according to the price list, and it signs the statement that commits the relevant laboratory to:
- follow the guidelines for each ICT and meet the overall schedule of ICT,
- perform all analyses in own laboratories by own staff,
- reimburse the costs of the ordered samples even when the samples are not received.

The completed application form must be delivered to the Water Research Institute by the specified deadline date.

Preparation, distribution and reception of samples

NRL pays particular attention to the proficiency testing in the entire PTS process, from taking natural samples through preparation of the synthetic and enriched natural samples and sample handling up to distribution of samples to participants in PTS. The provider complies with all conditions required for ensuring the quality of delivered samples and he follows the guidelines for sampling process; selection of suitable material for sample bottles/containers; methods of preservation, transport and storage of samples in accordance with the Slovak Technical Standards of the STN EN ISO 5667 series. The NRL has developed detailed standard operating procedures for the preparation of samples and PTS processes that are included in the activity specification.

Prepared samples are tested for homogeneity and stability where only respective proportion of the sample is analyzed, i.e. by random selection of sampling containers/bottles.

To prevent collusion between the participants the provider prepares some of the samples at different concentration levels and selectively distributes the samples. The participants are informed about this fact in advance.

Samples are distributed to all participants in one day within a single ICT and they are delivered to the three distribution centres - Bratislava, Banska Bystrica and Košice.

The provider also prepares duplicate samples for distribution centres (in case of any damage to samples or increasing the order for samples). In exceptional cases, the samples are shipped to participating laboratories via express mail companies (UPS, etc.) at their own expenses. Only preserved synthetic samples can be delivered in this way and the date of analyses applied to all ICT participants must be kept.

In the distribution centres, the samples are delivered to designated employees based on received instructions and the list of participants included in the acceptance protocol. Participants (qualified/competent employees) take samples personally against their signature together with supporting documentation (instructions for sample processing and data report forms)

The instructions for the ICT participants contain at least the information on methods for handling the samples (storage, pre-treatment, preparation for analysis and processing), a list of methods used, dates of analyzes and deadlines for result submission.

The Data report form is a document in which the results of analyses are recorded by participants. The Data report form includes the following:

- ICT identification/name,
- Participant identification – registration number, name of laboratory, contact information,
- The table of results (parameter, sampling container no., units, ID code of testing method, date of analysis, indicated uncertainties),
- Name and signature of the employee responsible for the results,
- Laboratory/company seal.

In the Data report form the participant can give comments and suggestions to the relevant ICT. The participant is required to make a statement that all analyzes were performed in the laboratory listed in the Data report form.

Selection of methods and techniques

When processing samples for ICT, the NRL as a provider of PTS requires the participants to proceed in the same manner as for regular samples analyzed in a laboratory by using established methods, unless otherwise stated in the instructions. Only in the specialized PTS aimed at comparison of methods and in several conventional methods it is required to use specific methods. The provider of PTS provides participants with a list of commonly available methods for different parameters together with a code assigned to a particular method. The provider requires the participants to identify a method in the Data report form, or to describe details of the technique or literature, when unconventional method is used. The final report of

the ICT includes the list and evaluation of the methods used for the determination of each parameter.

Evaluation of ICT Results

The methods of statistical evaluation are in accordance with the requirements of international documents [1], [4], [5]. Results submitted by the ICT participants are processed using the ISO 5725 software. Data are processed also in Excel, where the check calculations, stability and homogeneity tests, evaluation of methods and graphics evaluation of laboratory performance are carried out. The overall process of evaluating the results obtained from the ICT is included in the NRL standard operating procedures. An important step in assessing the results is the determination of reference values and evaluation criteria.

Determination of Reference Value

When determining the reference value the NRL proceeds in accordance with the rules specified in Section 3.2.7 of the Standard [6]. In addition, the NRL takes into account the requirements based on the method of sample preparation, sample type and required analyzes. According to the above facts, the NRL determines the following reference values:

- certified reference value (synthetic samples of basic chemical analysis, trace inorganic and organic analysis, radio-chemical analysis),
- verified known value based on specified sample preparation by diluting and mixing the reference material (synthetic samples of basic chemical analysis, trace inorganic analysis and radio-chemical analysis),
- consensus value based on the results of so-called reference laboratories (accredited laboratories with a long-time high success rate in the inter-laboratory comparison tests)

Accepted reference values are communicated to the participants in the preliminary results.

Determination of Evaluation Criteria

Determination of evaluation criteria, i.e. parameter determining the range (interval) of acceptable values, depends on several factors such as the concentration level of monitored parameter, the accuracy of methods as well as the results of homogeneity and stability tests. With regard to the reference value, the ICT provider uses mainly the following criteria for individual fields of testing:

Basic chemical, trace inorganic and radio-chemical analyses	± 10 to 25 %,
Trace organic analysis	± 20 to 50 %
Hydro-biological, microbiological and eco-toxicological analyses	± s_R to 2 s_R.

Evaluation of Inter-laboratory Comparison Tests

The evaluation of the ICT results is anonymous and particular results are known only to the organization processing the ICT results and to a participant. Participant is provided with information on results in three stages: preliminary results (only in the field of chemistry), the final report and the workshop "Evaluation of Inter-Laboratory Comparison Tests"

Preliminary results: The results can be found under the identification codes (rk). Information on identification code and the ICT results are received by participant in the so-called preliminary results, which includes a cover letter containing the table of accepted reference values of parameters.

Final report: It contains all the relevant information about ICT: objective and focus of MPS; dates; deadlines; list of participants; basic data on preparation, homogeneity and stability of samples; the methods used; reference values and interval of values appropriate for individual parameters; statistical evaluation of the results; a brief assessment of the results; and the statement of the provider on the performance of laboratories and on the comments and proposals of the ICT participants. The final report sent to every ICT participant includes the Certificate of performance of laboratory in interlaboratory comparison tests (the correctness of the results) as well as the appendix containing the results obtained in the test. The Final Report on the ICT results and the Certificate are delivered to participants by ordinary mail only after the payment of the cost for the ordered ICT.

The Workshop "Evaluation of Inter-Laboratory Comparison Tests" is prepared by the provider to conclude relevant ICT. Participation in the workshop is free of charge for each participant. The workshop is organized to present the results of a given ICT to the participants and provide them with information on the overall ICT preparation, related problems and comments of the participants. The ICT consulting days are equivalent of the workshop. The participants have the opportunity to discuss individually the obtained results, used methods, problems with sample analysis, the causes of failure in ICT, etc. during the consulting days.

If a participant raises objections to the organization, process or evaluation of inter-laboratory comparison tests, he may submit them in writing to the PTS Coordinator within 10 days of the date of the occurrence of the fact objected by him.

Conclusion

Inter-laboratory comparison tests are among fundamental components of the external quality control system. They provide laboratories with objective evidence of the level of reliability of the results produced by them. Based on the results obtained in the test, the ICT participants can verify or improve their quality system in the laboratory. Successful participation in the ICT enables laboratories to demonstrate their performance by the Certificate of Participation in ICT.

The precondition for participation in the ICT organized by NRL is the registration in the NRL Register. The registered participants should notify the provider of any change in address, phone, fax and contact persons. At the beginning of the year, the ICT provider sends an offer in the form of application for specific ICT to all participants registered in the NRL database.

References

[1] EN ISO/IEC 17043:2010, Conformity assessment – General requirements for proficiency testing
[2] ILAC-G13:07/2007, ILAC guidelines for the requirements for the competence of providers of proficiency testing schemes (before 2012)
[3] EN ISO/IEC 17025:2005, General requirements for the competence of testing and calibration laboratories
[4] ISO/FDIS 13528:2005, Statistical methods for use in proficiency testing by interlaboratory comparisons
[5] P ISO/TS 20612:2009, Water quality. Interlaboratory comparisons for proficiency testing of analytical chemistry laboratories
[6] ISO 3534-2:2006, Statistics. Vocabulary and symbols, Part 2 Applied statistics