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Accredited by Slovak National Accreditation Service for testing, Reg. No. 059/S-100,
and for providing Proficiency testing schemes, Reg. No. 059/T-005.
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Information on Proficiency Testing Schemes organised by the Slovak National Water Reference Laboratory

Introduction

The Slovak National Water Reference Laboratory (SNWRL) is part of the Water Research Institute (WRI). Currently is carrying out accredited and non-accredited activity. The non-accredited activity is not in conflict of interest with the area of proficiency testing schemes (PTS). The main objective of the proficiency testing scheme (PTS) is to ensure regular external check 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 of laboratories. With properly working feedback in the quality system, the proficiency testing allows laboratories to improve the quality of their performance and the results of analyses and enhances the credibility of laboratory performance.

Definitions/terms [1]

- Proficiency testing (PT) - evaluation of participant performance against pre-established 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 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 test item – synthetic sample to check the quality of water parameters at the concentration level of drinking/surface/waste water.
- Proficiency test 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. (In the case of sampling process, the participant of the proficiency test is the sampling group, which will sampling water samples in accordance with the requirements of Slovak technical standards methods for the type of water and additional requirements of the proficiency test provider.)

Competency of the SNWRL as a provider of PTS

The Water Research Institute (WRI) has been providing hydro-analytical laboratories in Slovakia with inter-laboratory comparison tests since 1987. SNWRL fulfils the requirements to prepare and implement PT, it has extensive experience with particular PT it is capable for measuring objects that have to be determined within the PT. This professional competency is accredited in accordance with the standard EN ISO/IEC 17025 [2]. The SNWRL's competency to perform the accredited work impartially and credibly is compliant with the requirements of EN ISO/IEC 17043 [1]. The specification of the activities of SNWRL as a provider of PTS is documented in the Certificate's Annex that includes the following:

- The scope of proficiency testing,
- Properties – compared parameters,

- Range of compared values,
- Proficiency testing repetition interval.

The subjects in terms of accreditation are divided in three areas:

- Area of chemical methodologies
- Area of biological methodologies
- Water sampling

The determined parameters are classified in the following groups based on the compared properties:

Area of chemical methodologies	• radio-chemical analysis (RR) • trace inorganic analysis (SAA) • trace organic analysis (SOA) • basic physical-chemical analysis (Z): for drinking/surface water (ZPV) for surface water/wastewater (ZOV)
Area of biological methodologies	• hydro-biological analysis (HBR) • microbiological analysis (MBR)
Water sampling	• sampling of drinking water (OPiV) • sampling of surface water (OPoV) • sampling of waste water (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 SNWRL is related to the analysis of drinking water, surface water and waste water in accordance with the requirements under effective legislation.

Samples for ICT are prepared and analysed in the accredited SNWRL testing laboratory [3].

During the water sampling PTS is besides the required water quality parameters determined on-site, are evaluated also techniques and methods, sampling devices, related documentation and test of theoretical knowledge. Samplings are carried out at selected sampling sites.

Participation in ICT organized by SNWRL

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 can apply for registration to the SNWRL Register by submitting a completed Application Form for Registration. For the confirmation of registration the applicant is sent a copy of confirmed application form.

The participation in PTS is anonymous and confidential. Participants are registered in the SNWRL database under the permanent registration number that is used for regular contact/communication with the ICT participants. In each ICT project the participants are assigned an identification code. It is a randomly generated number that changes 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.

Binding Registration Form for Inter-laboratory Comparison Test

At the beginning of each year, the SNWRL prepares an annual plan of proficiency testing scheme in which the following information is provided:

- Field of activity (chemical, biological methods, water sampling),
- 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 SNWRL 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 application submission, method of distribution, distribution centre, etc.

If interested, the laboratory shall indicate 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,
- cover the costs of the ordered samples even though the samples are not taken over.

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

Preparation, distribution and reception of samples

SNWRL 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 containers/bottles, methods of preservation, transport and storage of samples in accordance with the Technical Standards of water properties for water sampling. The SNWRL 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 while only respective proportion of the sample is analysed, i.e. by random selection of sampling containers/bottles.

To prevent secret agreement 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 in terms of a single ICT are distributed to all participants at the same day and they are delivered to the three distribution centres - Bratislava, Banská Bystrica and Košice. Foreign participants and, in exceptional cases, Slovak participants, do receive the samples by courier service at their own expenses. This way, only model and preserved samples can be distributed and the deadline must be met for the analyses which is valid for all the participants.

The provider also prepares duplicate (spare) samples for distribution centres (in case of any damage of samples or extended demand).

In the distribution centres, the samples are handed over to designated personnel based on registration forms and the list of participants included in the acceptance protocol. Participants (qualified/competent employees) take samples in person together with the supporting documentation (instructions for sample processing and data report forms) and sign the acceptance protocol.

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 analyses 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, laboratory name, contact information,
- the table of results (parameter, sampling number, units, ID code of testing method, date of analysis, indicated uncertainties and space for the participants notes on the performance of analyses),
- 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 analyses were performed in the laboratory listed in the Data Report Form. The completed and authorized Data Report Form has to be sent to the Water Research Institute (WRI) within the deadline.

Selection of methods and techniques

When processing samples for ICT, the SNWRL as a provider of PTS requires the participants to proceed in the same manner as for regular samples analysed in their laboratory by using routine 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 the 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 the international standards used in PTS [1]. Results submitted by the ICT participants are processed in PC with a valid software system. Data are processed in Excel in which the calculations, stability and homogeneity tests, evaluation of methods and graphics evaluation of laboratory performance are carried out. The overall process of the evaluation of 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 SNWRL proceeds in accordance with the rules specified in chapter 7 of Standards B.2 [1]. In addition, the SNWRL takes into account the requirements based on the method of sample preparation, sample type and required analyses. According to the above facts, the NRL determines the following reference values:

- Based on specific preparation of a sample by diluting or mixing certified or reference material (C)RM – **derived reference value**,
- **Certified reference value** when CRM is used,
- Based on participants' results - **consensus value** calculated based on the statistical methodology described in ISO 13528 [3].

Accepted reference values are first communicated to the participants on the website of the Water Research Institute (WRI) after the deadline of results submission.

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 regards to the reference value, the ICT provider uses mainly the following criteria in percentages for individual fields of testing:

- **For the area of chemical methodologies**

± from 10 to 40 % (RR, SAA, ZPV, ZOV);
± from 20 to 50 % (SOA);

- *For the area of biological methodologies:*

± from 1s_R to 3 s_R (HBR, MBR), while (s_R) = standard deviation of reproducibility of statistical complex

- **For water sampling are** the criteria for determination range of the satisfactory results of the sampling analyses derived in accordance with the requirements of the Governmental Regulations and Standards from the criteria appropriate for the monitored parameters in the concrete ICT.

Given the nature of the activities in terms of ICT focusing on water sampling the evaluation criteria of the sampling groups, in addition to the evaluation of the analyses of the selected required parameters on site and / or in the samples taken are: participants 'own sampling performance, sampling equipment, theoretical knowledge of the members on the executed type of sampling and assessment of the level of the required documentation used for the water samples.

Evaluation of Inter-laboratory Comparison Tests

The evaluation of the ICT results is anonymous and the particular results are known only to the processor of the ICT results and to the participant. The participant is informed about the results in the final ICT report. The participants will have a possibility to consult their results, applied methodologies,

issues with the analysis of samples, causes of unsuccessful ICT etc. directly with an NRL employee responsible for the specific PTS area.

Final report: Contains all relevant information about ICT - objective and focus of the ICT, deadlines, list of participants, basic information on sample preparation, their homogeneity and stability, the applied methodologies, reference values and range of satisfactory values for individual parameters, statistical evaluation of the results, brief evaluation of the results and the organizer's comments on the laboratory performance and the comments and suggestions of participants of the ICT. Part of the final report sent to each participant in water testing is their ***Certificate of accuracy of the results*** obtained and the Annex with the results achieved.

In the water sampling the groups after a successful participation in the ICT will receive two certificates: ***Certificate of participation in the ICT that includes the names of the members of the group*** and ***a Certificate of competency to carry out samplings for a specific type of water and of the accuracy of the results obtained in the respective ICT***. The final report on the results of ICT and the relevant certificates are sent to participants by email only after the pay-off of the costs for the ordered ICT.

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 30 days from the date of the occurrence of the objection.

The PTS coordinator's contact details:

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Conclusion

Inter-laboratory comparison tests belong to the 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.

References

[1] ISO/IEC 17043:2010, Conformity assessment – General requirements for proficiency testing
[2] ISO/IEC 17025:2018, General requirements for the competence of testing and calibration laboratories
[3] ISO 13528:2022, Statistical methods for use in proficiency testing by interlaboratory comparison