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Kakovost vode - Medlaboratorijske primerjave za ugotavljanje usposobljenosti analiznih kemijskih laboratorijev

Water quality - Interlaboratory comparisons for proficiency testing of analytical chemistry laboratories

Qualité de l'eau - Comparaisons interlaboratoires pour des essais de compétence de laboratoires de chimie analytique

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Water quality — Interlaboratory comparisons for proficiency testing of analytical chemistry laboratories

Qualité de l'eau — Comparaisons interlaboratoires pour des essais de compétence de laboratoires de chimie analytique

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Foreword

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In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of document:

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ISO/TS 20612 was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Introduction

Participation in interlaboratory tests in various test fields offers a testing laboratory an opportunity to obtain an objective picture of its proficiency. Such tests serve as a confidence-building measure both for the laboratory itself and for prospective clients.

This Technical Specification is based on the following international recognized documents:

— ISO/IEC Guides 43-1 and 43-2;

— ISO 13528;

— *The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories* – (IUPAC, ISO, AOAC);

— ILAC Guide 13;

— ISO/IEC 17025;

— ISO 5725-1 and ISO 5725-2.

As these documents only define a framework for design, execution and evaluation of proficiency testing by interlaboratory comparisons, this Technical Specification describes in detail an evaluation procedure which is especially suitable for the sector of water, waste water and sludge analysis, where results of interlaboratory comparisons play an important role in the admission of laboratories to certain analytical tasks. Therefore, the fairness of assessment of laboratories must be guaranteed. Assessment should not be dependent on the provider, the date, or the method of evaluation.

Water quality — Interlaboratory comparisons for proficiency testing of analytical chemistry laboratories

1 Scope

This Technical Specification specifies the criteria related to proficiency testing by interlaboratory comparisons in the field of water, waste water and sludge analysis. In particular, it specifies the requirements in respect to proficiency test providers and to the design, execution and evaluation of laboratory proficiency comparisons.

This document may be used if the determinands in the interlaboratory test may be regarded as capable of measurement with a certain degree of continuity. This is generally the case for chemical constituents and physicochemical determinands, but continuity does not always exist in the case of biological and/or microbiological determinands.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3534-1, *Statistics — Vocabulary and symbols — Part 1: General statistical terms and terms used in probability*

ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-1:1994/Cor.1:1998, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions — Technical Corrigendum 1*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 13528, *Statistical methods for use in proficiency testing by interlaboratory comparisons*

ISO/IEC Guide 43-1, *Proficiency testing by interlaboratory comparisons — Part 1: Development and operation of proficiency testing schemes*

ISO/IEC Guide 43-2, *Proficiency testing by interlaboratory comparisons — Part 2: Selection and use of proficiency testing schemes by laboratory accreditation bodies*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO/IEC 17020, *General criteria for the operation of various types of bodies performing inspection*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3534-1, ISO 5725-1, ISO 5725-2, ISO/IEC Guide 43-1, ISO/IEC Guide 43-2 and the following apply.

3.1
breakdown point
smallest percentage of outlier laboratories above which the estimation method may be entirely inapplicable

3.2
efficiency
ratio of the variance of the optimum estimation method for normal distribution to the variance of the estimation method under consideration, each assuming a normal distribution

NOTE This is expressed as a percentage.

3.3
sample
totality of a homogeneous analysis material with an identical composition or quality (similar to term batch)

3.4
subsample
defined portion of a sample obtained by suitable sample division and identical in terms of composition

4 Symbols

d_i Absolute difference between log-linear variance function and the logarithm of the reproducibility standard deviation

$G_1(x_i)$ Generalized distribution function of interlaboratory differences with continuity correction (s_R)

$G_2(x_i)$ Generalized distribution function of intralaboratory differences with continuity correction (s_r)

g Quality limit

$H_1(x_i)$ Generalized distribution function of interlaboratory differences (s_R)

$H_2(x_i)$ Generalized distribution function of intralaboratory differences (s_r)

i Index denoting the serial number of one of p samples

J_i Number of participants in the case of sample i

j Index denoting the serial number of one of J participating laboratories

k_1,k_2 Correction factors for calculating z_U-score

μ Overall mean

μ_i Overall mean of i-th sample

x_a Assigned value

n_j Number of measurements made by laboratory j

q	Quantile parameter
p	Number of samples (levels)
PG_0, PG_1	Testing values for testing variance function
s_R	Reproducibility standard deviation
s_{R_i}	Reproducibility standard deviation of i-th sample
$\hat{s}_{R_i}$	Reproducibility standard deviation for sample i adjusted using a variance function
s_r	Repeatability standard deviation
$\hat{\sigma}$	Standard deviation for proficiency assessment
W	Weighting matrix
X	Design matrix
X^T	Transposed design matrix
x_i	Discontinuity point
y	Arithmetic mean of test results of an unspecified laboratory
y_{ji}	Measurement result for the i-th measurement made by laboratory j
y_j	Arithmetic mean of measurement results of laboratory j
z	z-Score, i.e. standardized deviation of laboratory result from assigned value
z_U	Corrected z-score
Ψ	Function for determining the Hampel estimator
$\chi^2_{p-2;0,95}$	95 % chi-squared quantile for $p - 2$ degrees of freedom
Φ	Distribution function of standard normal distribution
α	Significance level
ν	Relative reproducibility standard deviation
γ	Vector of logarithms of standard deviations
θ_0, θ_1	Parameters of log-linear variance function
$\tilde{\theta}_0$	Estimate of logarithm of relative reproducibility standard deviation if independent from concentration level

5 Requirements relating to proficiency test provider

Proficiency testing by interlaboratory comparisons must lie in the responsibility of specialists who are familiar not only with the requirements relating to the design, execution and evaluation of interlaboratory tests, but also with the analytical methods to be tested, and who have demonstrated their specialist knowledge. Against this background, it is recommended that the test provider regularly organizes interlaboratory tests in the relevant test field.

The proficiency test provider must maintain an adequately documented quality management system based on the criteria specified in ISO/IEC 17020 or ISO/IEC 17025, covering all necessary framework conditions, responsibilities and standard operation procedures.

In addition all measurements within the framework of the provided proficiency test should fulfil the technical requirements as specified in ISO/IEC 17025.

An advisory group that includes specialists for all the fields involved should be appointed to enable the relevant interlaboratory test system to be brought into line with the state of the art and proper account to be taken of the specialist requirements relating to the interlaboratory tests. Keeping a written record of the group's decisions is recommended.

6 Participants

Only laboratories that have the requisite staffing and equipment for the tests to be performed shall take part in an interlaboratory test. Each participating laboratory should appoint a member of staff to be responsible for maintaining contact with the proficiency test manager and ensuring that the analyses are correctly carried out in accordance with the proficiency test manager's instructions.

7 Proficiency test design

7.1 Proficiency test plan

All details of the proficiency test design should be laid down in a plan prior to the start of the interlaboratory test. This includes especially details about:

— involved staff;

— sample matrix;

— determinands to be analysed;

— concentration level of the determinand;

— number of samples;

— sample containers;

— sample preservation;

— distribution of samples;

— communication with participants of the proficiency test (PT);

— homogenization method;

— homogeneity and stability check;