For Comments Only

Draft Indian Standard

Quality of Measurement Results — Criteria for Variation in Repeat/Replicate Testing

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FOREWORD

(Formal clauses to be added later on)

The quality of measurement results depends precisely on how accurate the measurement result is. In other words, how close the measurement result is to the true value or accepted reference value. It is also known that accuracy is not the only parameter that describes the quality of measurement result. Precision and the bias are also equally important in describing the quality of measurement result. But all these terms are valid only if the measurement process is in statistical control.

In case of repeat or replicate measurements using the same test method, there may be a different combination of precision and bias (in case of reproducible results) and depending on which combination of sources of variability affect the test results, it has to be worked out. For example, results obtained by one operator on the same (repeat) or similar samples (replicate tests) may give a different result, variation that can be attributed to different environmental control, even though homogeneity of specimen is ensured. Similarly, when other operators use same measurement process for the same or similar test specimen, variations in the measurement result may be attributed to environment, operator experience and training etc. Such situations are practically faced in everyday laboratory functioning. Repeat or replicated results on same or similar specimen may differ from operator to operator, due to the environment that would have affected bias and due to the training of the operator that would have affected the precision.

While developing/designing a test method, care is taken for the long term statistical stability of the test method. The studies are conducted to assess the various influencing parameters/variables that are believed to have major impact on the measurement results produced by that method and on precision and bias of that method.

Many sources affect variability in the measurement process. Only by controlling these sources, can the measurement process be controlled. The only necessity is the identification of these sources. Some of the sources are:

i. Experimental realization of test method
ii. Operator
iii. Apparatus
iv. Environment
v. Test specimen (sample)

vi. Time

The above list gives are some of the numerous factors that introduce variability in measurement results. It is however left to the operator himself to determine the factors that may introduce variability in the measurement, as he is the best person to judge the conditions under which measurements are taken. The best method could be to list out all the factors and negate some or many of these by making approximations and taking remedial actions. Other prominent factors (other than those given above) that introduce variability shall be counted and measurement shall be made alternatively by varying one or many of those factors and keeping other factors constant. The results thus obtained shall be collated and analyzed to know the effect of factors on measurement.

In case of repeat/replicate measurements, the factors that introduce variability in the original test and repeat/replicate tests may not be the same; hence, there may be variation in original repeated/replicated results. The limit to this variation can be attributed to the factors that caused such variation. In order to find the factors or the reasons of variation in original and repeated/replicated results, all the factors causing variability in measurement result should be accounted for.

Quality of Measurement Results — Criteria for Variation in Repeat/Replicate Testing

1. SCOPE:

This document establishes a criteria of variation in results of repeat/ replicated measurements, when measurements are made under conditions of repeatability and repeat measurements are made under conditions of reproducibility.

The criteria derived in this document can also be used to set limits of variation in the results of repeat/ replicated measurements.

2. NORMATIVE REFERENCES:

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

IS/ISO	**Title**
ISO 5725-1	Accuracy (trueness and precision) of measurement methods and results, Part 1: General principles and definitions.
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 5725-3	Accuracy (trueness and precision) of measurement methods and results, Part 3: Intermediate measures of the precision of a standard measurement method.
ISO 21748	Guidance for the use of repeatability, reproducibility and trueness estimates in Measurement uncertainty estimation.

3. TERMS AND DEFINITIONS:

The terms and definitions given in ISO 5725-1 shall apply.

4. SYMBOLS:

The terms and definitions given in ISO 5725-2 shall apply.

5. ESTIMATES OF PARAMETERS IN BASIC MODEL:

5.1. The procedure given in this document is based on the statistical model given in Clause 5 of ISO 5725-1:2023 and elaborated upon in ISO 5725-1:2023, 1.2. In particular, these procedures are based on formulae (2) to (6) if ISO 5725-1:2023, clause 5.
The model is

$$y = m + B + e \qquad \text{……..(1)}$$

Where

y is the measurement result
m is the general mean or the expected mean
B is the bias term (laboratory component of bias under repeatability conditions)
e is the random error occurring in every measurement under repeatability conditions.

5.2. Bias Term B

In case, the true value (μ) is known, the expected mean m is replaced by μ. Usually, the expected mean m is not equal to the true value, $m \neq \mu$.
Thus,

$$m - \mu = \delta$$

or

$$m = \mu + \delta,$$

δ being the bias of the measurement method.
Equation (1) can be rewritten as

$$y = \mu + \delta + B + e$$
$$y = \mu + \Delta + e \quad \ldots\ldots (2)$$

Equation (2) contains the intrinsic bias of measurement method (δ) and laboratory component of bias (B). Thus, total bias

$$\Delta = \delta + B$$

is a combination of method bias and laboratory component of bias and is a combination of random and systematic components.

Under repeatability conditions, for repeat measurements, the laboratory component of bias consists of biases due to several influencing factors, which influence the measurement process continuously. If B_0, B_1, B_2, etc. are the biases due to these influencing factors like operator, environment, calibration, temperature, etc., then the laboratory component of bias can be expressed as

$$B = B_0 + B_1 + B_2 \ldots \quad \ldots\ldots(3)$$

It is assumed that all these influencing factors are independent of each other.

When a test method is intended to test different levels of test specimen, e.g. a method is used to determine the diameter of different sizes of UPVC pipes or a test method is used to determine the different concentrations of an element in solution, then in such cases the effect of bias can be ignored as it may have no influence here. However, in situations when a method is used to test the same material/specimen repeatedly, as in the case of repeat/replicate testing, the effect of bias will be there and the effect on measurement result has to be evaluated. Thus, the variance in B is

$$Var(B) = \sigma_L^2,$$

where σ_L includes the between operator, between time and between equipment variability. σ_L is also known as between laboratory variance (ISO 5725-1).

If repeat/replicate testing is done with changes in the influencing factors, like different operators, different environmental conditions, different time at which measurements are made etc, intermediate variances can be calculated. Then the variance in bias *B* is considered to be composed of variances due to different components, given by $Var(B_0)$, $Var(B_1)$, $Var(B_2)$… If it is assumed that the distribution of laboratory component of bias is approximately normal, the variance of *B* is given as

$$Var(B) = Var(B_o) + Var(B_1) + Var(B_2) + ...$$

which includes all the terms/factors that can cause bias.

$$\sigma_L^2 = \sigma_0^2 + \sigma_1^2 + \sigma_2^2 + ...$$

When making comparison between two results using the same method at different times, by different operators, on different set of instruments/equipment, the laboratory component of bias for these terms (*B*) must be considered and determined (reproducibility). In practical situations, during any repeat measurement, the sample and even set of instruments/equipment may remain the same; the rest of the factors may vary.

5.3. The error term *e*

The error term *e* is due to the random error in the measurement and occurs in every measurement. It consists of all the random components of errors that can be present in a measurement. When the repeat measurements are made in a single laboratory, the variance in *e* can be termed as within laboratory variance, and denoted as:

$$Var(e) = \sigma_w^2 \qquad \text{……(4)}$$

For a single test under repeatability conditions, this error term is constant and its contribution in repeat/replicate testing is constant. σ_w is within laboratory variance (ISO 5725-1).

5.4. The deviation term

Apart from the effect of bias and error in the measurement result, there may remain some unaccounted factors that may introduce some deviations in the results of repeat testing. These factors are expressed by deviation term and incorporate all those terms/effects that have not been considered elsewhere. This is expressed as $\sum_i c_i x_i'$.

Where

x_i' is the deviation from the nominal value (expected mean *m*)

c_i is the sensitivity coefficient, equal to $\partial y/\partial xi$
Now, equation (2) can be rewritten as

$$y = \mu + \delta + B_0 + B_1 + B_2 + ... + e + \sum_i c_i x_i' \qquad ....(5)$$

For repeat measurements, the variation due to random effect can be nullified. The deviation term x_i' is very small and can be ignored. In such a case, equation (5) becomes

$$y = \mu + \delta + B_0 + B_1 + B_2 + ... + e \qquad ......(6)$$

A specimen differs from another specimen of the same class in two basic ways: the level of the measurand present and effect of other materials present in the material. The specimen is the totality of all characteristics (properties), other than the measurand, that have effect on the measured value. The effect of the error (precision) and bias will always be there but the combination of sources of error (precision) and bias will be different in different situations.

6. REPRODUCIBILITY VARIANCE

When estimating variance due to error term, the contribution of error term may vary due to different factors under repeatability like skills of operators etc. This value when averaged over repeated/replicated tests is called 'repeatability variance' and is designated as

$$\sigma_r^2 = [Var(e_1) + Var(e_2) + Var(e_3) + ... + Var(e_n)]/n,$$

where e_1, e_2, e_3 etc are 'n' different random error terms present in the measurement, independent of each other.

The reproducibility of a measurement method is due to the variation in the ever changing factors that affect the measurement result. If all the factors have been accounted in terms B and e, then 'Reproducibility Variance' is given as (see ISO 5725-1)

$$\sigma_R^2 = \sigma_L^2 + \sigma_r^2 \qquad ....(7)$$

where σ_r^2 is repeatability variance.

For repeat/replicate tests in a single laboratory

$$\sigma_r^2 = \sigma_w^2 \qquad ......(8)$$

Hence, from equations (7) and (8)

$$\sigma_R^2 = \sigma_L^2 + \sigma_w^2 \qquad ......(9)$$

or

$$\sigma_R = \sqrt{(\sigma_L^2 + \sigma_w^2)}$$

This value of 'σ_R', known as reproducibility variance, now encompasses all the effects/factors that can contribute to variability in measurement result due to various combinations of bias and error. It expresses the dispersion of the variation in measurement results under varying factors of bias and error contributors. This value can be used for criteria for fixing limits for repeat/replicate testing. It is expected that within a laboratory, the measurement result of specimen by a particular test method shall be within this limit if the measurement process is in state of statistical control. If it is not so, it implies that the measurement process is not in a state of statistical control and corrective action should be taken by the laboratory for it after proper root cause analysis.

7. LIMITS FOR REPEAT/ REPLICATE TESTING

In order to derive the limits for variability in the measurement results in repeat/replicate testing, it is first assumed that all the measurements follow normal distribution, unless there are reasons to believe otherwise. The limits for variation could be set as:

$$\text{Limits} = \pm 2\sigma_R \qquad \ldots\ldots(10)$$

in the coverage probability of approximately 95 percent. The result of repeat/replicate testing shall be within these limits. If required, the associated uncertainty can also be given in the limits, which is given by (using (6))

$$u^2(y) = u^2(\delta') + \sigma_R^2 \qquad \ldots\ldots(11)$$

where $u^2(y)$ is the uncertainty associated with the measurement result y,
$u^2(\delta')$ is uncertainty associated with δ,
σ_R is the reproducibility standard deviation

If the same method is used for determining measurement result, as in the case of repeat/replicate tests, the intrinsic bias of measurement method (δ) is constant, and hence $u(\delta')$=0, equation (11) can be rewritten as

$$u^2(y) = \sigma_R^2$$

From equation (10), Limits = $\pm 2u(y)$ ……(12)

in confidence interval of approximately 95 percent,

The limits given by equations (10) and (12) fix the limits for the criteria within which measurement results of repeat/replicate result should lie.

In order to determine the variability in repeat/replicate testing, repeated measurements are taken under conditions of repeatability. Measurements can also be taken by under varying the factors repeatedly so that the measurements are taken in all conditions. After all the measurements are taken, the limits are decided as above for within a single laboratory single test variability limit.

Many methods prescribe the limits of repeatability and reproducibility standard deviations. This indicates that the deviation from the mean of measurement results for a single sample by use of that method shall be within repeatability standard deviation 'σ_r' (under repeatability conditions) and it shall be within reproducibility standard deviation 'σ_R' (under reproducibility conditions). For repeat/replicate results using such methods, the value of reproducibility standard deviation 'σ_R' may be taken as limit under which the results of repeated/replicated measurements would fall.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Statistical Methods for Quality, Data Analytics and Reliability, MSD 03

Organization(s)	*Representative(s)*
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Bharat Electronics Ltd., Ghaziabad	Ms Ekta Bhardwaj
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