codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

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TO: Codex Contact Points
Interested International Organizations

FROM: Secretary, Codex Alimentarius Commission
Joint FAO/WHO Food Standards Programme
FAO, 00100 Rome, Italy

SUBJECT: **Analytical Terminology for Codex Use (Procedural Manual)**

DEADLINE: **20 September 2004**

COMMENTS: To:

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The 24th Session of the Codex Committee on Methods of Analysis and Sampling agreed to initiate the revision of the definitions contained in the Codex Procedural Manual (Analytical Terminology for Codex Use) (ALINORM 03/23, para. 95). This was approved by the 26th Session of the Commission as new work (ALINORM 03/41, para. 138-140 and Appendix VIII). The Twenty-Fifth session of the Codex Committee on Methods of Analysis and Sampling (ALINORM 04/27/23) agreed to develop a paper on definitions of analytical terminology for use in the Codex Procedural Manual. The intent is to present this paper for discussion at the 26th Session of the Committee on Methods of Analysis and Sampling (Budapest, Hungary, 4-8 April 2005).

Comments are, therefore, requested on both the current definitions of analytical terms and any suggestions for additional analytical terms that should be included in the Codex Procedural Manual. A 2002 compendium entitled “Harmonization of analytical terminology in accordance with international standards”, which includes Codex definitions and those from other international organizations, is included as **Annex 1**. This compendium was prepared to facilitate discussions on the harmonization of analytical terminology by the chairman of the Inter-agency Meeting on Methods of Analysis and Sampling. Modification of these definitions and additional terms and definitions not included in this document may be submitted for consideration.

Governments and international organizations wishing to provide comments and suggestions to be included in the paper, and for consideration at the next session of the Committee, should do so in writing, preferably by email, to the above addresses **before 20 September 2004**.

ANNEX 1

INTER-AGENCY MEETING

HARMONISATION OF ANALYTICAL TERMINOLOGY IN ACCORDANCE WITH INTERNATIONAL STANDARDS

INTRODUCTION

There has been frequent discussion in the Codex Alimentarius Commission (CAC) and other organisations with respect to definitions. Many users of analytical methods of analysis wish to have available to them a set of definitions, and for these to be available in a comparative form.

This paper, prepared under the auspices of the Inter-Agency Meeting (IAM), is an attempt to bring together some of the definitions which are of interest to members of the IAM, and to users of their standardised methods of analysis and sampling in a readily available comparative format.

They have been taken for a number of different sources, principally those adopted by the CAC itself, and the primary sources that were used when the CAC brought its set of definitions together. In addition, the definitions stipulated in the various Harmonised Guidelines/Protocols have been cited.

FORMAT

The definitions are identified as follows:

Title of definition

Organisations preparing the definition

Text of definition

The definitions are arranged alphabetically.

ACTION RECOMMENDED BY THE IAM

The Inter-Agency Meeting discussed definitions at its Fourteenth Session, held Budapest, February, 2001 and recommended that this comparative document be prepared and finalised under its auspices. The IAM members have therefore been requested to provide the definitions that their respective organisations have adopted.

Note: it is appreciated that there will be considerable duplication of a single definition for, say, trueness, but that will be readily identified. It is the differences that are more surprising.

It is also recognised that a considerable number of the definitions presently given are in need of revision: e.g. laboratory bias, fitness for purpose, empirical method.

It is also recognised that there are a considerable number of definitions to be entered, notably those connected with sampling, and indeed sampling within the laboratory, e.g. target, increment,

aggregate sample, laboratory sample, test portion, test solution, aliquot, etc.

Definitions Covered

DEFINITIONS ARE GIVEN FOR THE FOLLOWING FROM SOURCES OF DIRECT RELEVANCE TO PARTICIPANTS AT THE INTER-AGENCY MEETING:

ACCURACY
Analytical run
Analytical system
Assigned value
Applicability:
Bias
Bias of the measurement method
Certified reference material
Control material
Co-ordinator
Empirical method of analysis
Error
Fitness for purpose
Internal quality control
Interlaboratory study
Interlaboratory test comparisons
Laboratory bias
Laboratory component of bias
Laboratory-performance (proficiency) study
Laboratory sample
Limit of detection
Limit of determination
Material-certification study
Measurement uncertainty (uncertainty of measurement)
METHOD-PERFORMANCE STUDY
Native analyte
ONE-WAY ANALYSIS OF VARIANCE
Precision
Proficiency testing scheme
Quality assurance
Quality assurance programme/system
Rational method of analysis:
Recovery
Reference material
Relative uncertainty
REPEATABILITY
Repeatability conditions
Repeatability limit
Repeatability standard deviation
Repeatability relative standard deviation
Reproducibility
Reproducibility conditions
Reproducibility limit
REPRODUCIBILITY STANDARD DEVIATION
Reproducibility relative standard deviation
Result
Ruggedness
Run (analytical run)
Sensitivity
Specificity
Standard matching solution
Standard reference solution
Standard solution
Standard volumetric solution
Surrogate
Surrogate recovery
Target value for standard deviation
Test portion
Test sample
Testing laboratory
Traceability
True value
Trueness
Validated range

ACCURACY

ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)

Closeness of agreement between a test result and an accepted reference value.

NOTE The term accuracy, when applied to a set of test results, involves a combination of random components and a common systematic error or bias component.

As a concept:

CODEX ALIMENTARIUS COMMISSION

The closeness of agreement between the reported result and the accepted reference value.

Note:

The term accuracy, when applied to a set of test results, involves a combination of random components and a common systematic error or bias component. {ISO 3534-1} When the systematic error component must be arrived at by a process that includes random error, the random error component is increased by propagation of error considerations and is reduced by replication.

As a statistic:

CODEX ALIMENTARIUS COMMISSION

The closeness of agreement between a reported result and the accepted reference value. {ISO 3534-1}

Note:

Accuracy as a statistic applies to the single reported final test result; accuracy as a concept applies to single, replicate, or averaged value.

International vocabulary for basic and general terms in metrology

Harmonised guidelines for internal quality control in analytical chemistry laboratories

The international harmonised protocol for the proficiency testing of (chemical) analytical laboratories

Closeness of the agreement between the result of a measurement and a true value of the measurand.

Note 1. Accuracy is a qualitative concept.

Note 2. The term *precision* should not be used for *accuracy*.

ANALYTICAL RUN

See "Run".

ANALYTICAL SYSTEM

The harmonised guidelines for internal quality control in analytical chemistry laboratories

Range of circumstances that contribute to the quality of analytical data, including equipment, reagents, procedures, test materials, personnel, environment and quality assurance measures.

ASSIGNED VALUE

International harmonised protocol for the proficiency testing of (chemical) analytical laboratories

The value to be used as the true value by the proficiency test co-ordinator in the statistical treatment of results. It is the best available estimate of the true value of the analyte in the matrix.

APPLICABILITY:

CODEX ALIMENTARIUS COMMISSION

The analytes, matrices, and concentrations for which a method of analysis may be used satisfactorily to determine compliance with a Codex standard.

Note

In addition to a statement of the range of capability of satisfactory performance for each factor, the statement of applicability (scope) may also include warnings as to known interference by other analytes, or inapplicability to certain matrices and situations.

BIAS

Codex Alimentarius Commission

The difference between the expectation of the test results and an accepted reference value. {ISO 3534-1}

Notes:

1. Bias is the total systematic error as contrasted to random error. There may be one or more systematic error components contributing to bias. A larger systematic difference from the accepted reference value is reflected by a larger bias value. {ISO 3534-1}

2. When the systematic error component(s) must be arrived at by a process that includes random error, the random error component is increased by propagation of error considerations and reduced by replication.

1. ***Statistics, vocabulary and symbols - Part 1: Probability and general statistical terms, ISO 3534 -1: 1993***
2. Harmonised guidelines for internal quality control in analytical chemistry laboratories

Difference between the expectation of the test results and an accepted reference value.

NOTE:

Bias is a systematic error as contrasted to random error. There may be one or more systematic error components contributing to the bias. A larger systematic difference from the accepted reference value is reflected by a larger bias value.

BIAS OF THE MEASUREMENT METHOD

International harmonised protocol for the proficiency testing of (chemical) analytical laboratories

The difference between the expectation of test results obtained from all laboratories using that method and an accepted reference value.

NOTE:

One example of this in operation would be where a method purporting to measure the sulphur content of a compound consistently fails to extract all the sulphur, giving a negative bias to the measurement method. the bias of the measurement method is measured by the displacement of the average of results from a large number of different laboratories all using the same method. The bias of a measurement method may be different at different levels.

CERTIFIED REFERENCE MATERIAL

1. International vocabulary for basic and general terms in metrology, 2nd Edition, 1993, ISO, Geneva
2. ***Harmonised guidelines for internal quality control in analytical chemistry laboratories***
3. International harmonised protocol for the proficiency testing of (chemical) analytical laboratories

Reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes its traceability to an accurate realisation of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence.

CONTROL MATERIAL

Harmonised guidelines for internal quality control in analytical chemistry laboratories

Material used for the purposes of internal quality control and subjected to the same or part of the same measurement procedure as that used for test materials.

CO-ORDINATOR

International harmonised protocol for the proficiency testing of (chemical) analytical laboratories

The organisation with responsibility for co-ordinating all of the activities involved in the operation of a proficiency testing scheme.

EMPIRICAL METHOD OF ANALYSIS

HARMONISED GUIDELINES FOR THE USE OF RECOVERY INFORMATION IN ANALYTICAL MEASUREMENT

A method which determines a value that can be arrived at only in terms of the method *per se* and serves by definition as the only method for establishing the measurand. (Sometimes called "defining method of analysis".)

ERROR

1. International vocabulary for basic and general terms in metrology, 2nd Edition, 1993, ISO, Geneva
2. ***Harmonised guidelines for internal quality control in analytical chemistry laboratories***

Result of a measurement minus a true value of the measurand.

FITNESS FOR PURPOSE

Harmonised guidelines for internal quality control in analytical chemistry laboratories

Degree to which data produced by a measurement process enables a user to make technically and administratively correct decisions for a stated purpose.

INTERNAL QUALITY CONTROL

1. Harmonised guidelines for internal quality control in analytical chemistry laboratories
2. ***International harmonised protocol for the proficiency testing of (chemical) analytical laboratories***

Set of procedures undertaken by laboratory staff for the continuous monitoring of operation and the results of measurements in order to decide whether results are reliable enough to be released. IQC primarily monitors the batchwise accuracy of results on quality control materials, and precision on independent replicate analysis of test materials.

INTERLABORATORY STUDY

CODEX ALIMENTARIUS COMMISSION

A study in which several laboratories measure a quantity in one or more "identical" portions of homogeneous, stable materials under documented conditions, the results of which are compiled into a single document.

Note

The larger the number of participating laboratories, the greater the confidence that can be placed in the resulting estimates of the statistical parameters. The IUPAC-1987 protocol (Pure & Appl. Chem., 66, 1903-1911(1994)) requires a minimum of eight laboratories for method-performance studies.

INTERLABORATORY TEST COMPARISONS

International harmonised protocol for the proficiency testing of (chemical) analytical laboratories

Organisation, performance and evaluation of tests on the same items or materials on identical portions of an effectively homogeneous material, by two or more different laboratories in accordance with pre-determined conditions.

LABORATORY BIAS

International harmonised protocol for the proficiency testing of (chemical) analytical laboratories

The difference between the expectation of the test results from a particular laboratory and an accepted reference value.

LABORATORY COMPONENT OF BIAS

International harmonised protocol for the proficiency testing of (chemical) analytical laboratories

The difference between the laboratory bias and the bias of the measurement method.

NOTES:

1. The laboratory component of bias is specific to a given laboratory and the conditions of measurement within the laboratory, and also it may be different levels of the test.

2. The laboratory component of bias is relative to the overall average result, not the true or reference value.

LABORATORY-PERFORMANCE (PROFICIENCY) STUDY

CODEX ALIMENTARIUS COMMISSION

An interlaboratory study that consists of one or more measurements by a group of laboratories on one or more homogeneous, stable, test samples by the method selected or used by each laboratory. The reported results are compared with those from other laboratories or with the known or assigned reference value, usually with the objective of improving laboratory performance.

Notes

1. Laboratory-performance studies can be used to support accreditation of laboratories or to audit performance. If a study is conducted by an organisation with some type of management control over the participating laboratories -- organisational, accreditation, regulatory, or contractual -- the method may be specified or the selection may be limited to a list of approval or equivalent methods. In such situations, a single test sample is insufficient to judge performance. It is expected that the results from 1 of every 20 tests will be outside the value for the calculated mean ± twice the standard deviation, due solely to random fluctuations.

2. Sometimes a laboratory-performance study may be used to select a method of analysis that will be used in a method-performance study. If all laboratories, or a sufficiently large subgroup, of laboratories, use the same method, the study may also be interpreted as a method-performance study, provided that the samples cover the range of concentration of the analyte.

3. Separate laboratories of a single organisation with independent facilities, instruments, and calibration materials, are treated as different laboratories.

Protocol for the design, conduct and interpretation of method-performance studies (1995)

An interlaboratory study that consists of one or more analyses or measurements by a group of laboratories on one or more homogeneous, stable test items, by the method selected or used by each laboratory. The reported results are compared with those of other laboratories or with the known or assigned reference value, usually with the objective of evaluating or improving laboratory performance.

LABORATORY SAMPLE

ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)

Sample as prepared for sending to the laboratory and intended for inspection or testing.

LIMIT OF DETECTION

Quantitative determinations

Nordic Committee On Food Analysis

The amount or the content of an analyte corresponding to the lowest measurement signal which with a certain statistical confidence may be interpreted as indicating that the analyte is present in the solution/analytical sample, but not necessarily allowing exact quantification.

$LD = 3*S_a/b$ where LD is the limit of detection, S_a is the standard deviation of x blank results ($x > 20$) and b is the slope of the calibration curve/regression line.

Codex Alimentarius Commission (Volume 3)

The smallest measured concentration of an analyte from which it is possible to deduce the presence of the analysts in the test sample with acceptable certainty. This determination should consider matrix related interference's with an instrumental signal to noise (S/N) ratio greater than 5:1 or the concentration determined by a factor of 3 standard deviations of the signal response for blank tissue, whichever is less.

Qualitative determination

Nordic Committee On Food Analysis

The threshold concentration below which positive identification is unreliable.

It is recommended that about ten replicate determinations are carried out at each level. The threshold concentration, i.e. the lowest concentration at which the method gives reliable positive results defined as y positive results of y separate determinations ($10 \leq y$).

LIMIT OF DETERMINATION

Codex Alimentarius Commission (volume 2)

The lowest concentration of a pesticide residue or contaminant that can be identified and quantitatively measured in a specified food, agricultural commodity, or animal feed with an acceptable degree of certainty by a regulatory method of analysis.

Codex Alimentarius Commission (Volume 3)

The limit of quantitation corresponds to the smallest measured concentration of residue from endogenously incurred test material above which a determination of the analyte can be made with a specified degree of certainty to its accuracy and precision.

Nordic Committee On Food Analysis

The limit of quantification (LQ) (also called limit of determination) of an analytical procedure is the lowest amount of analyte in a sample which can be quantitatively determined with a certain confidence.

$LQ = 10*S_a/b$ where LQ is the limit of quantification, S_a is the standard deviation of x blank results ($x > 20$) and b is the slope of the calibration curve/regression line.

MATERIAL-CERTIFICATION STUDY

CODEX ALIMENTARIUS COMMISSION

An interlaboratory study that assigns a reference value ("true value") to a quantity (concentration or property) in the test material, usually with a stated uncertainty.

NOTE: A material-certification study often utilises selected reference laboratories to analyse a candidate reference material by a method(s) judged most likely to provide the least-biased estimates of concentration (or of a characteristic property) and the smallest associated uncertainty.

Protocol for the design, conduct and interpretation of method-performance studies (1995)

An interlaboratory study that assigns a reference value ('true value') to a quantity (concentration or property) in the test item, usually with a stated uncertainty.

MEASUREMENT UNCERTAINTY (UNCERTAINTY OF MEASUREMENT)

1. ***Guide to the expression of uncertainty in measurement,* ISO, Geneva, 1993**
2. ***Harmonised guidelines for internal quality control in analytical chemistry laboratories***

Parameter, associated with the result of a measurement, that characterises the dispersion of the values that could reasonably be attributed to the measurand.

NOTES:

1. The parameter may be, for example, a standard deviation (or a given multiple of it), or the half-width of an interval having a stated level of confidence.

2. Uncertainty of measurement comprises, in general, many components. Some of these components may be evaluated from the statistical distribution of results of a series of measurements and can be characterised by experimental standard deviations. The other components, which can also be characterised by standard deviations, are evaluated from assumed probability distributions based on experience or other information.

3. It is understood that the result of a measurement is the best estimate of the value of a measurand, and that all components of uncertainty, including those arising from systematic effects, such as components associated with corrections and reference standards, contribute to the dispersion.

METHOD-PERFORMANCE STUDY

CODEX ALIMENTARIUS COMMISSION

An interlaboratory study in which all laboratories follow the same written protocol and use the same test method to measure a quantity in sets of identical test samples. The reported results are used to estimate the performance characteristics of the method. Usually these characteristics are within-laboratory and among-laboratories precision, and when necessary and possible, other pertinent characteristics such as systematic error, recovery, internal quality control parameters, sensitivity, limit of determination, and applicability.

Notes

1. The materials used in such a study of analytical quantities are usually representative of materials to be analysed in actual practice with respect to matrices, amount of test component (concentration), and interfering components and effects. Usually the analyst is not aware of the actual composition of the test samples but is aware of the matrix.

2. The number of laboratories, number of test samples, number of determinations, and other details of the study are specified in the study protocol. Part of the study protocol is the procedure which provides the written directions for performing the analysis.

3. The main distinguishing feature of this type of study is the necessity to follow the same written protocol and test method exactly.

4. Several methods may be compared using the same test materials. If all laboratories use the same set of directions for each method and if the statistical analysis is conducted separately for each method, the study is a set of method-performance studies. Such a study may also be designated as a method-comparison study.

Protocol for the design, conduct and interpretation of method-performance studies (1995)

An interlabortory study in which all laboratories follow the same written protocol and use the same test method to measure a quantity in sets of identical test items [test samples, materials]. The reported results are used to estimate the performance characteristics of the method. Usually these characteristics are within-laboratory and among-laboratories precision, and when necessary and possible, other pertinent characteristics such as systematic error, recovery, internal quality control parameters, sensitivity, limit of determination, and applicability.

NATIVE ANALYTE

Harmonised guidelines for the use of recovery information in analytical measurement

Analyte incorporated into the test material by natural processes and manufacturing procedures(sometimes called “incurred analyte”). Native analyte includes incurred analyte and incurred residue as recognised in some sectors of the Analytical Community. It is so defined to distinguish from analyte added during the analytical procedure.

ONE-WAY ANALYSIS OF VARIANCE

Protocol for the design, conduct and interpretation of method-performance studies (1995)

One-way analysis of variance is the statistical procedure for obtaining the estimates of within-laboratory and between-laboratory variability on a material-by-material basis. Examples of the calculations for the single level and single-split-level designs can be found in ISO 5725-1986.

PRECISION

1. ***ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)***
2. **CODEX ALIMENTARIUS COMMISSION**

The closeness of agreement between independent test results obtained under stipulated conditions {ISO 3534-1}

Notes: {ISO 3534-1}

1. Precision depends only on the distribution of random errors and does not relate to the true value or to the specified value.

2. The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results. Less precision is reflected by a larger standard deviation.

3. “Independent test results” means results obtained in a manner not influenced by any previous result on the same or similar test object. Quantitative measures of precision depend critically on the stipulated conditions. Repeatability and reproducibility conditions are particular sets of extreme conditions.

1. TERMS AND DEFINITIONS USED IN CONNECTIONS WITH REFERENCE MATERIALS, ISO GUIDE 30:1992
2. Harmonised guidelines for internal quality control in analytical chemistry laboratories
3. The international harmonised protocol for the proficiency testing of (chemical) analytical laboratories

Closeness of agreement between independent test results obtained under prescribed conditions.

NOTES:

1. Precision depends only on the distribution of random errors and does not relate to the accepted reference value.

2. The measure of precision is usually expressed in terms of imprecision and computed as a standard deviation of the test results. High imprecision is reflected by a larger standard deviation.

3. 'Independent test results' means results obtained in a manner not influenced by any previous result on the same or similar material.

PROFICIENCY TESTING SCHEME

International harmonised protocol for the proficiency testing of (chemical) analytical laboratories

Methods of checking laboratory testing performance by means of interlaboratory tests.

[It includes comparison of a laboratory's results at intervals with those of other laboratories, with the main object being the establishment of trueness]

QUALITY ASSURANCE

1. QUALITY ASSURANCE AND QUALITY MANAGEMENT - VOCABULARY, ISO 8402:1994
2. Harmonised guidelines for internal quality control in analytical chemistry laboratories

All those planned and systematic actions necessary to provide adequate confidence that a product or service will satisfy given requirements for quality.

QUALITY ASSURANCE PROGRAMME/SYSTEM

International harmonised protocol for the proficiency testing of (chemical) analytical laboratories

The sum total of a laboratory's activities aimed at achieving the required standard of analysis. While IQC and proficiency testing are very important components a quality assurance programme must also include staff training, administrative procedures, management structure, auditing etc. Accreditation bodies judge laboratories on the basis of their quality assurance programme.

RATIONAL METHOD OF ANALYSIS:

Harmonised guidelines for the use of recovery information in analytical measurement

A METHOD WHICH DETERMINES AN IDENTIFIABLE CHEMICAL(S) OR ANALYTES(S). FOR WHICH THERE MAY BE SEVERAL EQUIVALENT METHODS OF ANALYSIS AVAILABLE.

RECOVERY

Harmonised guidelines for the use of recovery information in analytical measurement

Proportion of the amount of analyte, present in or added to the analytical portion of the test material, which is extracted and presented for measurement.

REFERENCE MATERIAL

1. ***International vocabulary for basic and general terms in metrology, 2nd Edition, 1993, ISO, Geneva***
2. ***Harmonised guidelines for internal quality control in analytical chemistry laboratories***
3. ***International harmonised protocol for the proficiency testing of (chemical) analytical laboratories***

MATERIAL OR SUBSTANCE ONE OF WHOSE PROPERTY VALUES ARE SUFFICIENTLY HOMOGENEOUS AND WELL ESTABLISHED TO BE USED FOR THE CALIBRATION OF AN APPARATUS, THE ASSESSMENT OF A MEASUREMENT METHOD, OR FOR ASSIGNING VALUES TO MATERIALS.

RELATIVE UNCERTAINTY

Harmonised guidelines for single-laboratory validation of methods of analysis

Uncertainty expressed as a relative standard deviation.

REPEATABILITY

1. ***ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)***
2. **CODEX ALIMENTARIUS COMMISSION**

Precision under repeatability conditions.

AOAC INTERNATIONAL

r = Repeatability, the value below which the absolute difference between 2 single test results obtained under repeatability conditions (i.e., same sample, same operator, same apparatus, same laboratory, and short interval of time) may be expected to lie within a specific probability (typically 95%) and hence $r = 2.8 \times s_r$.

REPEATABILITY CONDITIONS

1. ***ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)***
2. **CODEX ALIMENTARIUS COMMISSION**
3. "Statistics, vocabulary and symbols - Part 1: Probability and general statistical terms", ISO 3534 -1: 1993
4. ***Harmonised guidelines for internal quality control in analytical chemistry laboratories***

Conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time.

REPEATABILITY LIMIT

1. ***ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)***
2. **CODEX ALIMENTARIUS COMMISSION**

The value less than or equal to which the absolute difference between two test results obtained under repeatability conditions may be expected to be with a probability of 95%.

Notes

1. The symbol used is r. {ISO 3534-1}

2. When examining two single test results obtained under repeatability conditions, the comparison should be made with the repeatability limit

$$r = 2.8\ s_r.$$ {ISO 5725-6, 4.1.4}

3 When groups of measurements are used as the basis for the calculation of the repeatability limits (now called the critical difference), more complicated formulae are required that are given in ISO 5725-6:1994, 4.2.1 and 4.2.2.

Protocol for the design, conduct and interpretation of method-performance studies (1995)

When the mean of the values obtained from two single determinations with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time, lies within the range of the mean values cited in the Final Report, 4.0, the absolute difference between the two test results obtained should be less than or equal to the repeatability limit (r) [= 2.8 x s_r) that can generally be inferred by linear interpolation of s_r from the Report.

NOTE: This definition, and the corresponding definition for reproducibility limit, has been assembled from five cascading terms and expanded to permit application by interpolation to a test item whose mean is not the same as that used to establish the original parameters, which is the usual case in applying these definitions. The term 'repeatability [and reproducibility] limit' is applied specifically to a probability of 95% and is taken as 2.8 x s_r [or s_R]. The general term for this statistical concept applied to any measure of location (e.g., median) and with other probabilities (e.g., 99%) is 'repeatability [and reproducibility] critical difference.'

REPEATABILITY STANDARD DEVIATION

CODEX ALIMENTARIUS COMMISSION

The standard deviation of test results obtained under repeatability conditions. {ISO 3534-1}

Notes {ISO 3534-1}

1. It is a measure of the dispersion of the distribution of test results under repeatability conditions.

2. Similarly "repeatability variance" and "repeatability coefficient of variation" could be defined and used as measures of the dispersion of test results under repeatability conditions.

AOAC INTERNATIONAL

s_r = Standard deviation, calculated from results generated under repeatability conditions.

REPEATABILITY RELATIVE STANDARD DEVIATION

AOAC INTERNATIONAL

RSD_r = Relative standard deviation, calculated from results generated under repeatability conditions [(s_r / $\bar{x}$) x 100], where $\bar{x}$ is the average of results over all laboratories and samples.

REPRODUCIBILITY

1. ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)
2. **CODEX ALIMENTARIUS COMMISSION**

Precision under reproducibility conditions.

REPRODUCIBILITY CONDITIONS

1. ***ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)***
2. **CODEX ALIMENTARIUS COMMISSION**

Conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment. {ISO 3534-1}

Note

When different methods give test results that do not differ significantly, or when different methods are permitted by the design of the experiment, as in a proficiency study or a material-certification study for the establishment of a consensus value of a reference material, the term "reproducibility" may be applied to the resulting parameters. The conditions must be explicitly stated.

AOAC INTERNATIONAL

Reproducibility (R), the value below which the absolute difference between single test results obtained under reproducibility conditions (i.e., on identical material obtained by operators in different laboratories, using the standardised test method), may be expected to lie within a certain probability (typically 95%); $R = 2.8 \times s_R$.

REPRODUCIBILITY LIMIT

1. ***ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)***
2. **CODEX ALIMENTARIUS COMMISSION**

The value less than or equal to which the absolute difference between two test results obtained under reproducibility conditions may be expected to be with a probability of 95%.

Notes

1. The symbol used is R. {ISO 3534-1}

2. When examining two single test results obtained under reproducibility conditions, the comparison should be made with the reproducibility limit

   $R = 2.8 \, s_R$. {ISO 5725-6, 4.1.4}

3 When groups of measurements are used as the basis for the calculation of the reproducibility limits (now called the critical difference), more complicated formulae are required that are given in ISO 5725-6:1994, 4.2.1 and 4.2.2.

Protocol for the design, conduct and interpretation of method-performance studies (1995)

When the mean of the values obtained from two single determinations with the same method on identical test items in different laboratories with different operators using different equipment, lies within the range of the mean values cited in the Final Report, 4.0, the absolute difference between the two test results obtained should be less than or equal to the reproducibility limit (R) [$= 2.8 \times s_R$] that can generally be inferred by linear interpolation of s_R from the Report.

NOTE 1: When the results of the interlaboratory test make it possible, the value of r and R can be indicated as a relative value (e.g., as a percentage of the determined mean value) as an alternative to the absolute value.

NOTE 2: When the final reported result in the study is an average derived from more than a single value, i.e., k is greater than 1, the value for R must be adjusted according to the following formula before using R to compare the results of a single routine analyses between two laboratories.

$R' = \{R^2 + r^2 (1 - [1/k])\}^{1/2}$

Similar adjustments must be made for replicate results constituting the final values for s_R and RSD_R if these will be the reported parameters used for quality control purposes..

NOTE 3: The repeatability limit, r, may be interpreted as the amount within which two determinations should agree with each other within a laboratory 95% of the time. The reproducibility limit, R, may be interpreted as the amount within which two separate determinations conducted in different laboratories should agree with each other 95% of the time.

NOTE 4: Estimates of s_R can be obtained only from a planned, organised method-performance study; estimates of s_r can be obtained from routine work within a laboratory by use of control charts. For occasional analyses, in the absence of control charts, within-laboratory precision may be approximated as one half s_R (Pure and Appl. Chem., 62, 149-162 (1990) , Sec. I.3, Note.).

REPRODUCIBILITY STANDARD DEVIATION

CODEX ALIMENTARIUS COMMISSION

The standard deviation of test results obtained under reproducibility conditions. {ISO 3534-1}

Notes {ISO 3534-1}

3. It is a measure of the dispersion of the distribution of test results under reproducibility conditions.

4. Similarly “reproducibility variance” and “reproducibility coefficient of variation” could be defined and used as measures of the dispersion of test results under reproducibility conditions.

AOAC INTERNATIONAL

s_R = Standard deviation, calculated from results under reproducibility conditions.

REPRODUCIBILITY RELATIVE STANDARD DEVIATION

AOAC INTERNATIONAL

RSD_R = Relative standard deviation calculated from results generated under reproducibility conditions $[(s_R / \bar{x}) \times 100]$

RESULT

CODEX ALIMENTARIUS COMMISSION

The final value reported for a measured or computed quantity, after performing a measuring procedure including all subprocedures and evaluations. {IUPAC-1994}

Notes: {VIM}

1 When a result is given, it should be made clear whether it refers to:

-- the indication [signal]

-- the uncorrected result

-- the corrected result

and whether several values were averaged.

2 A complete statement of the result of a measurement includes information about the uncertainty of measurement.

RUGGEDNESS

CODEX ALIMENTARIUS COMMISSION

The ability of a chemical measurement process to resist changes in results when subjected to minor changes in environmental and procedural variables, laboratories, personnel, etc. {IUPAC-1995}

RUN (ANALYTICAL RUN)

Harmonised guidelines for internal quality control in analytical chemistry laboratories

Set of measurements performed under repeatability conditions.

SENSITIVITY:

CODEX ALIMENTARIUS COMMISSION

Change in the response divided by the corresponding change in the concentration of a standard (calibration) curve; i.e., the slope, s_i, of the analytical calibration curve.

Note

This term has been used for several other analytical applications, often referring to capability of detection, to the concentration giving 1% absorption in atomic absorption spectroscopy, and to ratio of found positives to known, true positives in immunological and microbiological tests. Such applications to analytical chemistry should be discouraged.

Notes {IUPAC-1987}

1. A method is said to be sensitive if a small change in concentration, c, or quantity, q, causes a large change in the measure, x; that is, when the derivative dx/dc or dx/dq is large.

Although the signal s_i may vary with the magnitude of c_i or q_i, the slope, s_i, is usually constant over a reasonable range of concentrations. s_i may also be a function of the c or q of other analytes present in the sample.

SPECIFICITY

CODEX ALIMENTARIUS COMMISSION

The property of a method to respond exclusively to the characteristic or analyte defined in the Codex standard.

Notes

1. Specificity may be achieved by many means: It may be inherent in the molecule (e.g., infrared or mass spectrometric identification techniques), or attained by separations (e.g., chromatography), mathematically (e.g., simultaneous equations), or biochemically (e.g., enzyme reactions). Very frequently methods rely on the absence of interferences to achieve specificity (e.g., determination of chloride in the absence of bromide and iodide).

2. In some cases specificity is not desired (e.g., total fat, fatty acids, crude protein, dietary fibre, reducing sugars).

STANDARD MATCHING SOLUTION

ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)

Solution of which the relevant characteristic is known or defined (for example colour, turbidity) and is used to assess the test solution in relation to that characteristic.

NOTE 1 The English term standard matching solution is used solely as a generic term for these solutions, and each solution is normally defined more precisely by the appropriate adjective (for example, “standard colorimetric solution2, “standard turbidimetric solution”).

NOTE 2 It may be prepared from solutions mentioned above or other solutions having the required characteristic.

NOTE 3 The method of preparation of standard matching solutions is normally given in the subclause “Calibration” (see A.13.7).

STANDARD REFERENCE SOLUTION

ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)

Solution used as a reference solution for calibrating other solutions.

NOTE 1 It is either prepared from a primary standard or calibrated by some other means.

NOTE 2 Many standard reference solutions which can be used to prepare standard solutions are commercially available.

STANDARD SOLUTION

ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)

Solution of accurately known concentration of an element, an ion, a compound or a group derived from the substance used for its preparation.

STANDARD VOLUMETRIC SOLUTION

ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)

Solution for titrimetric analysis, the concentration of which is known accurately.

SURROGATE

Harmonised guidelines for the use of recovery information in analytical measurement

Pure compound or element added to the test material, the chemical and physical behaviour of which is taken to be representative of the native analyte.

SURROGATE RECOVERY

HARMONISED GUIDELINES FOR THE USE OF RECOVERY INFORMATION IN ANALYTICAL MEASUREMENT

Recovery of a pure compound or element specifically added to the test portion or test material as a spike. (Sometimes called "marginal recovery".)

TARGET VALUE FOR STANDARD DEVIATION

International harmonised protocol for the proficiency testing of (chemical) analytical laboratories

A numerical value for the standard deviation of a measurement result, which has been designated as a goal for measurement quality.

TEST PORTION

ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)

The quantity of material drawn from the test sample (or from the laboratory sample if both are the same) and on which the test or observation is actually carried out.

TEST SAMPLE

ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)

Sample prepared from the laboratory sample and from which test portions will be taken.

TESTING LABORATORY

International harmonised protocol for the proficiency testing of (chemical) analytical laboratories

A laboratory that measures, examines, tests, calibrates or otherwise determines the characteristics or performance of materials or products.

TRACEABILITY

1. International vocabulary for basic and general terms in metrology, 2nd Edition, 1993, ISO, Geneva
2. ***Harmonised guidelines for internal quality control in analytical chemistry laboratories***

Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties.

TRUE VALUE

International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories

The actual concentration of the analyte in the matrix.

TRUENESS

CODEX ALIMENTARIUS COMMISSION

The closeness of agreement between the average value obtained from a series of test results and an accepted reference value.

Notes

1. The measure of trueness is usually expressed in terms of bias. {ISO 3534-1}

2. Trueness has been referred to as “accuracy of the mean”.

1. ***Statistics, vocabulary and symbols - Part 1: Probability and general statistical terms, ISO 3534 -1: 1993***
2. ***Harmonised guidelines for internal quality control in analytical chemistry laboratories***
3. ***International harmonised protocol for the proficiency testing of (chemical) analytical laboratories***

Closeness of the agreement between the average value obtained from a large series of test results and an accepted reference value.

NOTE - The measure of trueness is usually expressed in terms of bias.

VALIDATED RANGE

Harmonised guidelines for single-laboratory validation of methods of analysis

That part of the concentration range of an analytical method which has been subjected to validation.

REFERENCES - (To be completed)

1. Codex Alimentarius Commission

2. Guide to the expression of uncertainty in measurement, ISO, Geneva, 1993

3. "International vocabulary for basic and general terms in metrology", 2nd Edition, 1993, ISO, Geneva

4. The Harmonised Guidelines For Internal Quality Control In Analytical Chemistry Laboratories, Pure Appl. Chem 1995, 67, 649 – 666

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6. ***The International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories, Pure Appl. Chem., 1993, 65, 2123-2144.***

7. International vocabulary for basic and general terms in metrology, 2nd Edition, 1993, ISO, Geneva

8. Nordic Committee On Food Analysis

9. ***Protocol For The Design, Conduct And Interpretation Of Method-Performance Studies (1995)***

10. *QUALITY ASSURANCE AND QUALITY MANAGEMENT - VOCABULARY, ISO 8402:1994*

11. Statistics, vocabulary and symbols - Part 1: Probability and general statistical terms, ISO 3534 -1: 1993

12. Terms and definitions used in connections with reference materials, ISO Guide 30:1992

13. ***ISO Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999)***