GUIDELINES ON ANALYTICAL TERMINOLOGY

(CAC/GL 72-2009)

INTRODUCTION

The Codex Committee on Methods of Analysis and Sampling has agreed on Analytical Terminology for Codex Alimentarius and government use. A number of these terms were previously included in the Codex Procedural Manual. In most cases terms used in the Procedural Manual were adopted over time with an underlying hierarchy and can be traced verbatim to specific editions of ISO 3534, the GUM, the VIM, the IUPAC Orange Book or other international standards already adopted by Codex. Definitions of terms that have changed with newer editions of the international standards from which they were originally adopted have been updated preserving the original hierarchy found in the Procedural Manual. In cases where terms have been added in addition to those originally found in the procedural manual an effort has been made to preserve the conceptual continuity and relationship of the newer terms with extant ones. These terms, together with the terms which are included in specific International Protocols/Guidelines already adopted by Codex by reference are given below.

ANALYTICAL TERMS

The following analytical terms are defined below:

Accuracy

Analyte

Applicability

Bias

Calibration

Certified reference material

Conventional quantity value

Critical value

Defining (Empirical) method of analysis

Error

Expanded measurement uncertainty

Fitness for purpose

HorRat

Inter-laboratory study

Laboratory performance (Proficiency) study

Limit of detection

Limit of quantification

Linearity

Material certification study

Measurand

Measurement method

Measurement procedure

Measurement uncertainty

Method-performance study
Metrological Traceability
Outlier
Precision
Quality assurance
Rational method of analysis
Recovery/recovery factors
Reference material
Reference value
Repeatability (Reproducibility)
Repeatability conditions
Repeatability (Reproducibility) limit
Repeatability (Reproducibility) standard deviation
Repeatability (Reproducibility relative standard deviation
Reproducibility conditions
Result
Robustness (ruggedness)
Selectivity
Sensitivity
Surrogate
Systematic error
Trueness
True value
Validated range
Validated Test Method
Validation
Verification

DEFINITIONS OF ANALYTICAL TERMS

Accuracy: The closeness of agreement between a test result or measurement result and a reference value.

Notes:

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

When applied to a test method, the term accuracy refers to a combination of trueness and precision.

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 2: Applied Statistics, ISO, Geneva, 2006

Analyte: The chemical substance sought or determined in a sample.

Note:

This definition does not apply to molecular biological analytical methods.

Reference:

Codex Guidelines on Good Laboratory Practice in Residue Analysis (CAC/GL 40-1993)

Applicability: the analytes, matrices, and concentrations for which a method of analysis may be used satisfactorily.

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.

Reference:

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

Bias: The difference between the expectation of the test result or measurement result and the true value. In practice conventional quantity value (VIM, 2007) can be substituted for true value.

Notes:

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.

The bias of a measuring instrument is normally estimated by averaging the error of indication over the appropriate number of repeated measurements. The error of indication is the: "indication of a measuring instrument minus a true value of the corresponding input quantity".

Expectation is the expected value of a random variable, e.g. assigned value or long term average {ISO 5725-1}

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 2: Applied Statistics, ISO, Geneva, 2006

Calibration: Operation that, under specified conditions, in a first step, establishes a relation between the values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and in a second step uses this information to establish a relation for obtaining a measurement result from an indication.

Notes:

A calibration may be expressed by a statement, calibration function, calibration diagram, calibration curve, or calibration table. In some cases it may consist of an additive or multiplicative correction of the indication with associated measurement uncertainty.

Calibration should not be confused with adjustment of a measuring system often mistakenly called "self calibration," or with verification of calibration.

Often the first step alone in the above definition is perceived as being calibration.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Certified reference material (CRM): Reference material accompanied by documentation issued by an authoritative body and providing one or more specified property values with associated uncertainties and traceability, using valid procedures

Notes:

Documentation is given in the form of a "certificate" (see ISO guide 30:1992).

Procedures for the production and certification of certified reference materials are given, e.g. in ISO Guide 34 and ISO Guide 35.

In this definition, "uncertainty" covers both measurement uncertainty and uncertainty associated with the value of the nominal property, such as for identity and sequence. Traceability covers both metrological traceability of a value and traceability of a nominal property value.

Specified values of certified reference materials require metrological traceability with associated measurement uncertainty {Accred. Qual. Assur., 2006}

ISO/REMCO has an analogous definition {Accred. Qual. Assur., 2006} but uses the modifiers metrological and metrologically to refer to both quantity and nominal properties.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

New definitions on reference materials, Accreditation and Quality Assurance, 10:576-578, 2006

Conventional quantity value: quantity value attributed by agreement to a quantity for a given purpose.

Notes:

The term "conventional true quantity value" is sometimes used for this concept, but its use is discouraged.

Sometimes a conventional quantity value is an estimate of a true quantity value.

A conventional quantity value is generally accepted as being associated with a suitably small measurement uncertainty, which might be zero.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Critical value (L_C): The value of the net concentration or amount the exceeding of which leads, for a given error probability α, to the decision that the concentration or amount of the analyte in the analyzed material is larger than that in the blank material. It is defined as:

$\Pr(\hat{L} > L_C \mid L=0) \leq \alpha$

Where $\hat{L}$ is the estimated value, L is the expectation or true value and L_C is the critical value.

Notes:

The definition of critical value is important for defining the Limit of Detection (LOD).

The critical value L_c is estimated by

$L_C = t_{1-\alpha\nu} s_o$,

Where $t_{1-\alpha\nu}$ is Student's-t, based on ν degrees of freedom for a one-sided confidence interval of $1-\alpha$ and s_o is the sample standard deviation.

If L is normally distributed with known variance, i.e. $\nu = \infty$ with the default α of 0.05, $L_C = 1.645 s_o$.

A result falling below the L_C triggering the decision "not detected" should not be construed as demonstrating analyte absence. Reporting such a result as "zero" or as < LOD is not recommended. The estimated value and its uncertainty should always be reported.

References:

ISO Standard 11843: Capability of Detection-1, ISO, Geneva, 1997

Nomenclature in evaluation of analytical methods, IUPAC, 1995

Defining (empirical/conventional) method of analysis: A method in which the quantity measured is defined by the result found on following the stated procedure.

Notes:

Empirical methods are used for purposes that cannot be covered by rational methods.

Bias in empirical methods is conventionally zero.

Reference:

Harmonised guidelines for single-laboratory validation of methods of analysis, 2002

Error: Measured quantity value minus a reference quantity value.

Note:

The concept of measurement 'error' can be used both: when there is a single reference value to refer to, which occurs if a calibration is made by means of a measurement standard with a measured value having a negligible measurement uncertainty or if a conventional value is given, in which case the measurement error is not known and if a measurand is supposed to be represented by a unique true value or a set of true values of negligible range, in which case the measurement error is not known.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Expanded measurement uncertainty: product of a combined standard measurement uncertainty and a factor larger than the number one

Notes:

The factor depends upon the type of probability distribution of the output quantity in a measurement model and on the selected coverage probability.

The term factor in this definition refers to a coverage factor.

Expanded measurement uncertainty is also termed expanded uncertainty.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

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

Reference:

Eurachem Guide: The fitness for purpose of analytical methods: A laboratory guide to method validation and related topics, 1998

HorRat: The ratio of the reproducibility relative standard deviation to that calculated from the Horwitz equation,

Predicted relative standard deviation $(PRSD)_R = 2C^{-0.15}$:

$HorRat(R) = RSD_R/PRSD_R$,

$HorRat(r) = RSD_r/PRSD_R$

Where C is concentration expressed as a mass fraction (both numerator and denominator expressed in the same units).

Notes:

The HorRat is indicative of method performance for a large majority of methods in chemistry.

Normal values lie between 0.5 and 2. (To check proper calculation of $PRSD_R$, a C of 10^{-6} should give a $PRSD_R$ of 16 %.)

If applied to within-laboratory studies, the normal range of HorRat(r) is 0.3-1.3.

For concentrations less than 0.12 mg/kg the predicted relative standard deviation developed by Thompson (The Analyst, 2000), 22% should be used.

References:

A simple method for evaluating data from an inter-laboratory study, J AOAC, 81(6):1257-1265, 1998

Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing, The Analyst, 125:385-386, 2000

Inter-laboratory study: 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.

Notes:

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.

Reference:

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

Laboratory-performance (proficiency) study: An inter-laboratory 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:

Laboratory-performance studies can be used to support laboratory accreditation of laboratories or to audit performance. If a study is conducted by an organization with some type of management control over the participating laboratories: organizational, accreditation, regulatory or contractual, the method may be specified or the selection may be limited to a list of approved or equivalent methods. In such situations, a single test sample is insufficient to judge performance.

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 test samples cover the range of concentration of the analyte.

Laboratories of a single organization with independent facilities, instruments, and calibration materials, are treated as different laboratories.

Reference:

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

Limit of Detection (LOD): The true net concentration or amount of the analyte in the material to be analyzed which will lead, with probability (1-β), to the conclusion that the concentration or amount of the analyte in the analyzed material is larger than that in the blank material. It is defined as:

$\Pr(\hat{L} \leq L_C \mid L=LOD) = \beta$

Where $\hat{L}$ is the estimated value, L is the expectation or true value and L_C is the critical value.

Notes:

The limit of detection LOD is estimated by,

$LOD \approx 2t_{1-\alpha\nu}\sigma_o$ [where $\alpha = \beta$],

Where $t_{1-\alpha\nu}$ is Student's-t, based on ν degrees of freedom for a one-sided confidence interval of 1-α and σ_o is the standard deviation of the true value (expectation).

$LOD = 3.29\ \sigma_o$, when the uncertainty in the mean (expected) value of the blank is negligible, $\alpha = \beta = 0.05$ and L is normally distributed with known constant variance. However, LOD is not defined simply as a fixed coefficient (e.g. 3, 6, etc.) times the standard deviation of a pure solution background. To do so can be extremely misleading. The correct estimation of LOD must take into account degrees of freedom, α and β, and the distribution of L as influenced by factors such as analyte concentration, matrix effects and interference.

This definition provides a basis for taking into account exceptions to simple case that is described, i.e. involving non-normal distributions and heteroscedasticity (e.g. "counting" (Poisson) processes as those used for real time PCR).

It is essential to specify the measurement process under consideration, since distributions, σ's and blanks can be dramatically different for different measurement processes.

At the limit of detection, a positive identification can be achieved with reasonable and/or previously determined confidence in a defined matrix using a specific analytical method.

References:

ISO Standard 11843: Capability of Detection-1, ISO, Geneva, 1997

Nomenclature in evaluation of analytical methods, IUPAC, 1995

Guidance document on pesticide residue analytical methods, Organization for Economic Cooperation and Development, 2007

Limit of Quantification (LOQ): A method performance characteristic generally expressed in terms of the signal or measurement (true) value that will produce estimates having a specified relative standard deviation (RSD), commonly 10% (or 6%). LOQ is estimated by:

$LOQ = k_Q\ \sigma_Q,\ k_Q = 1/RSD_Q$

Where LOQ is the limit of quantification, σ_Q is the standard deviation at that point and k_Q is the multiplier whose reciprocal equals the selected RSD. (The approximate RSD of an estimated σ, based on ν-degrees of freedom is $1/\sqrt{2\nu}$.)

Notes:

If σ is known and constant, then $\sigma_Q = \sigma_o$, since the standard deviation of the estimated quantity is independent of concentration. Substituting 10% in for k_Q gives:

$LOQ = (10 * \sigma_Q) = 10\ \sigma_o$

In this case, the LOQ is just 3.04 times the limit of detection, given normality and $\alpha = \beta = 0.05$

At the LOQ, a positive identification can be achieved with reasonable and/or previously determined confidence in a defined matrix using a specific analytical method.

This definition provides a basis for taking into account exceptions to the simple case that is described, i.e. involving non-normal distributions and heteroscedasticity (e.g. "counting" (Poisson) processes as those used for real time PCR).

References:

Nomenclature in evaluation of analytical methods, IUPAC, 1995

Guidance document on pesticide residue analytical methods, Organization for Economic Co-operation and Development, 2007

Linearity: The ability of a method of analysis, within a certain range, to provide an instrumental response or results proportional to the quantity of analyte to be determined in the laboratory sample. This proportionality is expressed by an *a priori* defined mathematical expression. The linearity limits are the experimental limits of concentrations between which a linear calibration model can be applied with an acceptable uncertainty.

Reference:

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

Material-Certification Study: An inter-laboratory 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 utilizes 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.

Reference:

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

Measurand: Quantity intended to be measured.

Notes:

The specification of a measurand requires knowledge of the kind of quantity, description of the state of the substance carrying the quantity, including any relevant component and the chemical entities involved.

In chemistry, 'analyte' or the name of a substance or compound are terms sometime used for measurand. This usage is erroneous because these terms do not refer to quantities.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Measurement method: Generic description of a logical organization of operations used in a measurement.

Note:

Measurement methods may be qualified in various ways such as: substitution measurement method, differential measurement method, and null measurement method; or direct measurement method, and indirect measurement method.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Measurement procedure: Detailed description of a measurement according to one or more measurement principles and to a given measurement method, based on a measurement model and including any calculation to obtain a result.

Notes:

A measurement procedure is usually documented in sufficient detail to enable an operator to perform a measurement.

A measurement procedure can include a statement concerning a target measurement uncertainty.

A measurement procedure is sometimes called a standard operating procedure (SOP).

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Measurement uncertainty: Non-negative parameter characterizing the dispersion of the values being attributed to a measurand, based on the information used.

Notes:

Measurement uncertainty includes components arising from systematic effects, such as components associated with corrections and the assigned values of measurement standards, as well as the definitional uncertainty. Sometimes estimated systematic effects are not corrected for but, instead associated measurement uncertainty components are incorporated.

The parameter may be, for example, a standard deviation called standard measurement uncertainty (or a given multiple of it), or the half-width of interval having a stated coverage probability.

Measurement uncertainty comprises, in general many components. Some of these components may be evaluated by Type A evaluation of measurement uncertainty from the statistical distribution of the values from a series of measurements and can be characterized by experimental standard deviations. The other components which may be evaluated by Type B evaluation of measurement uncertainty can also be characterized by standard deviations, evaluated from assumed probability distributions based on experience or other information.

In general, for a given set of information, it is understood that the measurement uncertainty is associated with a stated quality value attributed to the measurand. A modification of this value results in a modification of the associated uncertainty.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Method-Performance Study: An inter-laboratory 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 quantification, and applicability.

Notes:

The materials used in such a study of analytical quantities are usually representative of materials to be analyzed 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.

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.

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

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.

Reference:

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

Metrological Traceability: Property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the stated measurement uncertainty.

Notes:

A reference can be a definition of a measurement unit through its practical realization, or a measurement procedure including the measurement unit for a non-ordinal quantity, or a measurement standard.

Metrological traceability requires an established calibration hierarchy.

Specification of the reference must include the time at which this reference was used in establishing the calibration hierarchy, along with any other relevant metrological information about the reference, such as when the first calibration in the calibration hierarchy was performed.

For measurements with more than one input quantity each of the input values should itself be traceable and the calibration hierarchy involved may form a branched structure or network. The effort involved in establishing the metrological traceability for each input value should be commensurate with its relative contribution to the measurement result.

Metrological traceability of a measurement result does not ensure that the measurement uncertainty is adequate for a given purpose or that there is an absence of mistakes.

A comparison between two measurement standards may be viewed as a calibration if the comparison is used to check and if necessary correct the value and measurement uncertainty of the measurement standards.

The ILAC considers the elements for confirming metrological to be an unbroken metrological traceability chain to an international measurement standard or a national measurement standard, a documented procedure, accredited technical competence, metrological to the SI and calibration intervals (see ILAC P-10:2002)

The abbreviated term 'traceability' is sometimes used to mean 'metrological traceability' as well as other concepts, such as sample traceability or document traceability or instrument traceability or material traceability, where history (trace) is meant. Therefore the full term of metrological traceability is preferred if there is any risk of confusion.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Harmonized guidelines for internal quality control in analytical chemistry laboratories, 1995

ILAC P-10, 2002

Outlier: A member of a set of values which is inconsistent with other members of that set

Note:

The following practice is recommended for dealing with outliers.

a) Tests such as Cochran's or Grubb's tests are applied to identify stragglers or outliers:
   - if the test statistic is less than or equal to its 5 % critical value, the item tested is accepted as correct;
   - if the test statistic is greater than its 5 % critical value and less than or equal to its 1 % critical value, the item tested is called a straggler and is indicated by a single asterisk;
   - if the test statistic is greater than its 1 % critical value, the item is called a statistical outlier and is indicated by a double asterisk.

b) It is next investigated whether the stragglers and/or statistical outliers can be explained by some technical error, for example:
   - a slip in performing the measurement,
   - an error in computation,
   - a simple clerical error in transcribing a test result,
   - analysis of the wrong sample.

   Where the error was one of the computation or transcription type, the suspect result should be replaced by the correct value; where the error was from analyzing a wrong sample, the result should be placed in its correct cell. After such correction has been made, the examination for stragglers or outliers should be repeated. If the explanation of the technical error is such that it proves impossible to replace the suspect

test result, then it should be discarded as a "genuine" outlier that does not belong to the experiment proper.

c) When any stragglers and/or statistical outliers remain that have not been explained or rejected as belonging to an outlying laboratory, the stragglers are retained as correct items and the statistical outliers are discarded unless the statistician for good reason decides to retain them.

References:
ISO Standard 5725-1: Accuracy (trueness and precision) of measurement methods and results Part 1: General principles and definitions, ISO, Geneva, 1994

ISO Standard 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, Geneva, 1994

Precision: The closeness of agreement between independent test/measurement results obtained under stipulated conditions.

Notes:

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

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.

Quantitative measures of precision depend critically on the stipulated conditions. Repeatability and reproducibility conditions are particular sets of extreme conditions.

Intermediate conditions between these two extreme conditions are also conceivable, when one or more factors within a laboratory (intra-laboratory e.g. the operator, the equipment used, the calibration of the equipment used, the environment, the batch of reagent and the elapsed time between measurements) are allowed to vary and are useful in specified circumstances.

Precision is normally expressed in terms of standard deviation.

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 2: Applied Statistics, ISO, Geneva, 2006

ISO Standard 5725-3: Accuracy (trueness and precision) of measurement methods and results Part 3: Intermediate measures of the precision of a standard measurement method, ISO, Geneva, 1994

Quality assurance: All those planned and systematic actions necessary to provide adequate confidence that analytical results will satisfy given requirements for quality.

Reference:

Harmonized guidelines for internal quality control in analytical chemistry laboratories, 1995

***Rational method of analysis*:** A method that determines an identifiable chemical(s) or analytes(s) for which there may be several equivalent methods of analysis available.

Reference:

Harmonized guidelines for the use of recovery information in analytical measurement, 1998

ISO/IEC Guide 17025:2005: General requirements for the competence of calibration and testing laboratories, ISO, Geneva, 2005

Recovery/recovery factors: Proportion of the amount of analyte, present in, added to or present in and added to the analytical portion of the test material, which is presented for measurement.

Notes:

Recovery is assessed by the ratio $\boldsymbol{R = C_{obs} / C_{ref}}$ of the observed concentration or amount $\boldsymbol{C_{obs}}$ obtained by the application of an analytical procedure to a material containing analyte at a reference level $\boldsymbol{C_{ref}}$.

C_{ref} will be: (a) a reference material certified value, (b) measured by an alternative definitive method, (c) defined by a spike addition or (d) marginal recovery.

Recovery is primarily intended for use in methods that rely on transferring the analyte from a complex matrix into a simpler solution, during which loss of analyte can be anticipated.

Reference:

Harmonized guidelines for the use of recovery information in analytical measurement, 1998

Use of the terms "recovery" and "apparent recovery" in analytical procedures, 2002

Reference material: Material, sufficiently homogeneous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process or in examination of nominal properties.

Notes:

Examination of a nominal property provides a nominal property value and associated uncertainty. This uncertainty is not a measurement uncertainty.

Reference materials with or without assigned values can be used for measurement precision control whereas only reference materials with assigned values can be used for calibration and measurement trueness control.

Some reference materials have assigned values that are metrologically traceable to a measurement unit outside a system of units. In a given measurement, a given reference material can only be used for either calibration or quality assurance.

The specification of a reference material should include its material traceability, indicating its origin and processing. {Accred. Qual. Assur., 2006}

ISO/REMCO has an analogous definition that uses the term measurement process to mean examination which covers both measurement of a quantity and examination of a nominal property.

References:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

New definitions on reference materials, Accred. Qual. Assur., 10:576-578, 2006

Reference value: Quantity value used as a basis of comparison with values of quantity of the same kind.

Notes:

A reference quantity value can be a true quantity value of a measurand, in which case it is unknown, or a conventional quantity value in which case it is known.

A reference quantity value with an associated measurement uncertainty is usually provided with reference to

a) a material, e.g. a certified reference material

b) a reference measurement procedure

c) a comparison of measurement standards.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Repeatability (Reproducibility): Precision under repeatability (reproducibility) conditions.

Reference:

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

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

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

AOAC International methods committee guidelines for validation of qualitative and quantitative food microbiological official methods of analysis, 2002.

Repeatability conditions: Observation conditions where independent test/measurement results are obtained with the same method on identical test/measurement items in the same test or measuring facility by the same operator using the same equipment within short intervals of time.

Note:

Repeatability conditions include: the same measurement procedure or test procedure; the same operator; the same measuring or test equipment used under the same conditions; the same location and repetition over a short period of time.

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 2: Applied Statistics, ISO, Geneva, 2006

Repeatability (Reproducibility) limit: The value less than or equal to which the absolute difference between final values, each of them representing a series of test results or measurement results obtained under repeatability (reproducibility) conditions may be expected to be with a probability of 95%.

Notes:

The symbol used is r [R]. {ISO 3534-2}

When examining two single test results obtained under repeatability (reproducibility) conditions, the comparison should be made with the repeatability (reproducibility) limit, r [R] = 2.8σr[R]. {ISO 5725-6, 4.1.4}

When groups of measurements are used as the basis for the calculation of the repeatability (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.

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 2: Applied Statistics, ISO, Geneva, 2006

ISO 5725-6 "Accuracy (trueness and precision) of a measurement methods and results—Part 6: Use in practice of accuracy value", ISO, 1994

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

***Repeatability (reproducibility) standard deviation*:** Standard deviation of test results or measurement results obtained under repeatability (reproducibility) conditions.

Notes:

It is a measure of the dispersion of the distribution of the test or measurement results under repeatability (reproducibility) conditions.

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 2: Applied Statistics, ISO, Geneva, 2006

Repeatability (reproducibility) relative standard deviation (coefficient of variation): Repeatability (reproducibility) standard deviation divided by the mean.

$RSD_{r[R]}$ is computed by dividing the repeatability (reproducibility) standard deviation by the mean.

Notes:

Relative standard deviation (RSD) is a useful measure of precision in quantitative studies.

This is done so that one can compare variability of sets with different means. RSD values are independent of the amount of analyte over a reasonable range and facilitate comparison of variabilities at different concentrations.

The result of a collaborative test may be summarized by giving the RSD for repeatability (RSDr) and RSD for reproducibility (RSD_R).

The RSD is also known as coefficient variation.

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 1: General statistical terms used in probability, ISO, Geneva, 2006

AOAC International methods committee guidelines for validation of qualitative and quantitative food microbiological official methods of analysis, 2002.

***Reproducibility conditions*:** Observation conditions where independent test/measurement results are obtained with the same method on identical test/measurement items in different test or measurement facilities with different operators using different equipment.

Reference:

ISO Standard 3534-2: Vocabulary and Symbols Part 2: Applied Statistics, ISO, Geneva, 2006

Result: Set of values being attributed to a measurand together with any other available relevant information

Notes:

A result of measurement generally contains 'relevant information' about the set of values, such that some may be more representative of the measurand than others. This may be expressed in the form of a probability density function.

A result of measurement is generally expressed as a single measured value and a measurement uncertainty. If the measurement uncertainty is considered to be negligible for some purpose, the measurement result may be expressed as a single measured value. In many fields, this is the common way of expressing a measurement result.

In the traditional literature and in the previous edition of the VIM, result was defined as a value attributed to a measurand and explained to mean an indication or an uncorrected result or a corrected result according to the context.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

***Robustness (ruggedness*):** A measure of the capacity of an analytical procedure to remain unaffected by small but deliberate variations in method parameters and provides an indication of its reliability during normal usage

Reference:

ICH Topic Q2 Validation of Analytical Methods, the European Agency for the Evaluation of Medicinal Products: ICH Topic Q 2 A - Definitions and Terminology (CPMP/ICH/381/95), 1995

Harmonized guidelines for single laboratory validation of methods of analysis, Pure and Appl. Chem., 2002

Selectivity: Selectivity is the extent to which a method can determine particular analyte(s) in a mixture(s) or matrice(s) without interferences from other components of similar behaviour.

Note:

Selectivity is the recommended term in analytical chemistry to express the extent to which a particular method can determine analyte(s) in the presence other components. Selectivity can be graded. The use of the term specificity for the same concept is to be discouraged as this often leads to confusion.

Reference:

Selectivity in analytical chemistry, IUPAC, Pure Appl Chem, 2001

Codex Alimentarius Commission, Alinorm 04/27/23, 2004

Codex Alimentarius Commission, Procedural Manual, 17th Edition, 2007

Sensitivity: Quotient of the change in the indication of a measuring system and the corresponding change in the value of the quantity being measured.

Notes:

The sensitivity can depend on the value of the quantity being measured

The change considered in the value of the quantity being measured must be large compared with the resolution of the measurement system.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Surrogate: 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.

Reference:

Harmonized guidelines for the use of recovery information in analytical measurement, 1998

Systematic error: Component of measurement error that in replicate measurements remains constant or varies in a predictable manner.

Notes:

A reference value for a systematic error is a true quantity value, or a measured value of a measurement standard of negligible measurement uncertainty, or a conventional value.

Sytematic error and its causes can be known or unknown. A correction can be applied to compensate for a known systematic error.

Systematic error equals measurement error minus random measurement error.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Trueness: The closeness of agreement between the average of an infinite number of replicate measured quantity values and a reference quantity value.

Note 1: Measurement trueness is not a quantity and thus cannot be expressed numerically, but measures for closeness of agreement are given in ISO 5725.

Note 2: Measurement trueness is inversely related to systematic measurement error, but is not related to random measurement error.

Note 3: Measurement accuracy should not be used for 'measurement trueness' and vice versa.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

True value: Quantity value consistent with the definition of a quantity.

Notes:

In the error approach to describing measurement, a true quantity value is considered unique and in practice unknowable. The uncertainty approach is to recognize that, owing to the inherently incomplete amount of detail in the definition of quantity, there is not a single true quantity value, but rather a set of quantity values consistent with the definition of a quantity. However, this set of values is, in principle and in practice unknowable. Other approaches dispense altogether with the concept of true quantity value and rely on the concept of metrological compatibility of measurement results for assessing their validity.

When the definitional uncertainty associated with the measurand is considered to be negligible compared to the other components of the measurement uncertainty the measurand may be considered to have an essentially “unique” true value.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Validation: Verification, where the specified requirements are adequate for an intended use.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

Validated Test Method: An accepted test method for which validation studies have been completed to determine the accuracy and reliability of this method for a specific purpose.

Reference:

ICCVAM Guidelines for the nomination and submission of new, revised and alternative test methods, 2003

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

Reference:

Harmonized guidelines for single-laboratory validation of methods of analysis, 2002

Verification: Provision of objective evidence that a given item fulfils specified requirements.

Notes:

When applicable method uncertainty should be taken into consideration.

The item may be e.g. a process, measuring procedure, material, compound or measuring system.

The specified requirement may be that a manufacturer's specifications are met.

Verification in legal metrology, as defined in VIM and in conformity assessment in general pertains to the examination and marketing and/or issuing of a verification certificate for a measuring system.

Verification should not be confused with calibration. Not every verification is a validation.

In chemistry, verification of the identity of the entity involved or of the activity, requires a description of the structure and properties of that entity or activity.

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VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

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