Elemental Analysis Manual

for Food and Related Products

The following is a section of the Elemental Analysis Manual for Food and Related Products.

For additional information and to view other sections of the manual, visit the Elemental Analysis Manual for Food and Related Products web page at http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm2006954.htm.

U.S. Department of Health & Human Services

FDA U.S. Food and Drug Administration

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GLOSSARY

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Accuracy: The closeness of agreement between a test result and an accepted reference value. When applied to test results, accuracy includes a combination of random and systematic error. When applied to test method, accuracy refers to a combination of trueness and precision. See also Trueness and Section 3.2.1

Action level: Level of concern or target level for an analyte that must be reliably identified or quantified in a sample.

Analyte: The chemical substance measured and/or identified in a test sample by the method of analysis.

Analytical batch: An analytical batch consists of samples, standards, and blanks which are analyzed together with the same method sequence and same lots of reagents and with the manipulations common to each sample within the same time period (usually within one day) or in continuous sequential time periods. See also Section 3.2.2

ASDL: Analytical Solution Detection Limit. The lowest level that can be detected in a test solution obtained after a test portion is digested. See also Limit of Detection and Section 3.2.1

ASQL: Analytical Solution Quantification Limit. The level in a test solution obtained after a test portion is digested that corresponds with the defined quantitation specification and converts to LOQ. See also Limit of Quantitation and Section 3.2.1

Bias: The difference between the expectation of the test result and the true value or accepted reference value. Bias is the total systematic error, and there may be one or more systematic error components contributing to the bias.

Blank: A substance that does not contain the analytes of interest and is subjected to the usual measurement process. Blanks can be further classified as method blanks, matrix blanks, reagent blanks, instrument blanks, and field blanks. See also FMB, Matrix Blank, MBK, MBK_L, and MBK_C and Section 3.2.1

Calibration: Determination of the relationship between the observed analyte signal generated by the measuring/detection system and the quantity of analyte present in the sample measured. Typically, this is accomplished through the use of calibration standards containing known amounts of analyte.

Calibration Standard: A known amount or concentration of analyte used to calibrate the measuring/detection system. May be matrix matched for specific sample matrices.

Carryover: Residual analyte from a previous sample or standard which is retained in the analytical system and measured in subsequent samples. Also called *memory*.

Check Analysis: Result from a second independent analysis which is compared with the result from the initial analysis. Typically, check analyses are performed by a different analyst using the same method.

Confirmation of Identity: Unambiguous identification of an analyte(s) by a highly specific technique such as mass spectrometry or by demonstration of results from two or more independent analyses in agreement.

Confirmatory Analysis/Method: Independent analysis/method used to confirm the result from an initial or screening analysis. A different method is often used in confirmation of screening results.

CRM: Certified Reference Material. Reference material accompanied by documentation (certificate) issued by an authoritative body and providing one or more specified property values with associated uncertainties and traceability, using valid procedures. Note: Standard Reference Material (SRM) is the trademark name of CRMs produced and distributed by the National Institute of Standards and Technology (NIST). See also Reference Material and Section 3.5

CS: Check solution. Solution with analytes at known concentrations that is analyzed periodically during and at the end of an analytical run. See section 3.2.4

Cut-off Concentration: In qualitative analysis, the concentration of the analyte that is either statistically lower than the level of concern (for limit tests) or at which positive identification ceases (for confirmation of identity methods). See also *Threshold Value*.

CVAAS: Cold Vapor-Atomic Absorption Spectrometry. See, for example, Method 4.5

DF: Dilution Factor. Factor by which the mass fraction (or concentration) in a diluted analytical solution is multiplied to obtain the mass fraction (or concentration) in the analytical solution. See also Section 3.4.3

False Negative Rate: In qualitative analysis, a measure of how often a test result indicates that an analyte is not present, when, in fact, it is present or, is present in an amount greater than a threshold or designated cut-off concentration.

False Positive Rate: In qualitative analysis, a measure of how often a test result indicates that an analyte is present, when, in fact, it is not present or, is present in an amount less than a threshold or designated cut-off concentration.

FAP: Fortified Analytical Portion. Analytical portion that was fortified (spiked) with analyte before digestion. The FAP is used to determine if the preparation procedure or sample matrix contribute bias to the analytical result. See also Section 3.2.4

FAS: Fortified Analytical Solution. Analytical solution that is fortified (spiked) with analyte(s) before instrumental determination of analyte concentration. The FAS is used to determine the need for further dilution of the analytical solution to account for matrix effects. See also Section 3.2.4

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.

FMB: Fortified Method Blank. MBK that was fortified (spiked) with analyte(s) before digestion. The FMB is used to determine if the fortification and analysis methodology is in control. See also Section 3.2.4

GFAAS: Graphite Furnace-Atomic Absorption Spectrometry. See, for example, Methods 4.2 and 4.3

Guidance Level: Level of concern or action level issued under good guidance practices that must be reliably identified or quantified in a sample.

ICPAES: Inductively Coupled Plasma-Atomic Emission Spectrometry. See, for example, Methods 4.4 and 4.6

ICPMS: Inductively Coupled Plasma-Mass Spectrometry. See, for example, Methods 4.7, 4.8, 4.10, and 4.11

ICS: Independent Check Solution. Solution with analytes at known concentrations prepared in-house or obtained from a source external to the laboratory and different from the source used for instrument standardization. See section 3.2.4

I**DL:** Instrumental Detection Limit. The lowest level that an instrument's detector can measure. It represents an ideal case (e.g., without matrix effect), LOD and ASDL apply to a real-world sample

Incurred Samples: Samples that contain the analyte(s) of interest, which were not derived from laboratory fortification but from sources such as exogenous exposure or endogenous origin. Exogenous exposure includes, for example, pesticide use, consumption by an animal, or environmental exposure.

Interference: A positive or negative response or effect on response produced by a substance other than the analyte. Includes spectral, physical, and chemical interferences which result in a less certain or accurate measurement of the analyte.

Intermediate Precision: Within-laboratory precision obtained under variable conditions, e.g., different days, different analysts, and/or different instrumentation.

Internal Standard: A chemical added to the sample, in known quantity, at a specified stage in the analysis to facilitate quantitation of the analyte. Internal standards are used to correct for matrix effects, incomplete spike recoveries, etc. Analyte concentration is deduced from its response relative to that produced by the internal standard. The internal standard should have similar physico-chemical properties to those of the analyte.

Laboratory Fortified Matrix: See *Matrix Spike*.

LDR: Linear Dynamic Range. The linear portion of a response curve.

Level of Concern: Level of concern is the concentration of an analyte in a sample that has to be exceeded before the sample can be considered violative. This concentration can be a regulatory tolerance, safe level, action level, guidance level or a laboratory performance level.

Limit Test: A type of semi-quantitative screening method in which analyte(s) has a defined level of concern. Also referred to as binary or pass/fail tests.

Linearity: Also called "linearity". The ability of a method, within a certain range, to provide an instrumental response or test results proportional to the quantity of analyte to be determined in the test sample.

LOD: Limit of Detection. The minimum amount or concentration of analyte that can be reliably distinguished from zero. The term is usually restricted to the response of the detection system and is often referred to as the *Detection Limit*. When applied to the complete analytical method it is often referred to as the *Method Detection Limit* (MDL). See 3.2.1

LOQ: Limit of Quantitation. The minimum amount or concentration of analyte in the test sample that can be quantified with acceptable precision. Limit of quantitation (or quantification) is variously defined but must be a value greater than the MDL and should apply to the complete analytical method. See 3.2.1

Matrix: All the constituents of the test sample with the exception of the analyte.

Matrix Blank: A substance that closely matches the samples being analyzed with regard to matrix components. Ideally, the matrix blank does not contain the analyte(s) of interest but is subjected to all sample processing operations including all reagents used to analyze the test samples. The matrix blank is used to determine the absence of significant interference due to matrix, reagents and equipment used in the analysis.

Matrix Effect: An influence of one or more components from the sample matrix on the measurement of the analyte concentration or mass. Matrix effects may be observed as increased or decreased detector responses, compared with those produced by simple solvent solutions of the analyte.

Matrix Source: The origin of a test matrix used in method validation. A sample matrix may have variability due to its source. Different food matrix sources can be defined as different commercial brands, matrices from different suppliers, or in some cases different matrices altogether. For example, if a variety of food matrices with differing physical and chemical properties are selected, the number of sources for each food sample matrix may be one or more.

Matrix spike: An aliquot of a sample prepared by adding a known amount of analyte(s) to a specified amount of matrix. A matrix spike is subjected to the entire analytical procedure to establish if the method is appropriate for the analysis of a specific analyte(s) in a particular matrix. Also referred to as a *Laboratory Fortified Matrix*.

MBK: Method blank. A substance that does not contain the analyte(s) of interest but is subjected to all sample processing operations including all reagents used to analyze the test samples. An aliquot of reagent water is often used as a method blank in the absence of a suitable analyte-free matrix blank. MBK_L is laboratory MBK and MBK_C is the MBK critical value. See 3.2.1

MCF: Mass Correction Factor. See 3.4.6

MDC: Minimum Detectable Concentration. In qualitative analysis, an estimate of the minimum concentration of analyte that must be present in a sample to ensure at a specified high probability (typically 95% or greater) that the measured response will exceed the detection threshold, leading one to correctly conclude that an analyte is present in the sample.

MDL: Method Detection Limit. The minimum amount or concentration of analyte in the test sample that can be reliably distinguished from zero. MDL is dependent on sensitivity, instrumental noise, blank variability, sample matrix variability, and dilution factor.

Method Development: The process of design, optimization and preliminary assessment of the performance characteristics of a method.

Method Validation: The process of demonstrating or confirming that a method is suitable for its intended purpose. Validation includes demonstrating performance characteristics such as accuracy, precision, specificity, limit of detection, limit of quantitation, linearity, range, ruggedness and robustness.

Method Verification: The process of demonstrating that a laboratory is capable of replicating a validated method with an acceptable level of performance.

m_o : Characteristic Mass. In graphite furnace atomic absorption spectrometry, the mass of analyte that produces an integrated absorbance signal of 0.0044 A-sec (or 0.0044 absorbance if peak signal). See also Section 3.2.1

PD: Percent Difference. See Section 3.4.5

Precision: The closeness of agreement between independent test results obtained under specified conditions. The precision is described by statistical methods such as a standard deviation or confidence limit of test results. See also *Random Error*. Precision can be further classified as *Repeatability, Intermediate Precision*, and *Reproducibility*. See 3.2.1

Qualitative Analysis/Method: Analysis/method in which substances are identified or classified on the basis of their chemical, biological or physical properties. The test result is either the presence or absence of the analyte(s) in question.

Quantitative Analysis/Method: Analysis/method in which the amount or concentration of an analyte may be determined (or estimated) and expressed as a numerical value in appropriate units with acceptable accuracy and precision.

Random error: Component of measurement error that in replicate measurements varies in an unpredictable manner. See also *Precision*.

Range: The interval of concentration over which the method provides suitable accuracy and precision.

Reagent Blank: Reagents used in the procedure taken through the entire method. Reagent Blanks are used to determine the absence of significant interference due to reagents or equipment used in the analysis.

Recovery: The proportion of analyte (incurred or added) remaining at the point of the final determination from the analytical portion of the sample measured. Usually recovery is expressed as a percentage.

RM: Reference material. A material, sufficiently homogenous 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. See also Certified Reference Material and Section 3.5

Reference standard: A standard, generally having the highest metrological quality available at a given location in a given organization, from which measurements are made or derived. Note: Generally, this refers to recognized national or international traceable standards provided by a standards producing body such as the National Institute of Standards and Technology (NIST).

Repeatability: Precision obtained under observation conditions where independent test results are obtained with the same method on identical test items in the same test facility by the same operator using the same equipment within short intervals of time. See 3.2.1

Representative Analyte: An analyte used to assess probable analytical performance with respect to other analytes having similar physical and/or chemical characteristics. Acceptable data for a representative analyte are assumed to show that performance is satisfactory for the represented analytes. Representative analytes should include those for which the worst performance is expected. Representative analytes are used mostly for non-targeted analysis and unknown screening procedures.

Representative Matrix: Matrix used to assess probable analytical performance with respect to other matrices, or for matrix-matched calibration, in the analysis of broadly similar commodities. For food matrices, similarity is usually based on the amount of water, fats, protein, and carbohydrates. Sample pH and salt content can also have a significant effect on some analytes.

Reproducibility: Precision obtained under observation conditions where independent test results are obtained with the same method on identical test items in different test facilities with different operators using different equipment. See 3.2.1

RPD - Relative Percent Difference: See Section 3.4.5

Ruggedness/Robustness: 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.

Screening Analysis/Method: An analysis/method intended to detect the presence of analyte in a sample at or above some specified concentration (action or target level). Screening methods typically attempt to use simplified methodology for decreased analysis time and increased sample throughput.

Selectivity: The extent to which a method can determine particular analyte(s) in a mixture(s) or matrix(ces) without interferences from other components of similar behavior. Selectivity is generally preferred in analytical chemistry over the term *Specificity*.

Sensitivity: The change in instrument response which corresponds to a change in the measured quantity (*e.g.*, analyte concentration). Sensitivity is commonly defined as the gradient of the response curve or slope of the calibration curve at a level near the LOQ.

Specificity: In quantitative analysis, specificity is the ability of a method to measure analyte in the presence of components which may be expected to be present. The term *Selectivity* is generally preferred over Specificity.

Spike Recovery: The fraction of analyte remaining at the point of final determination after it is added to a specified amount of matrix and subjected to the entire analytical procedure. Spike Recovery is typically expressed as a percentage. Spike recovery should be calculated for the method as written. For example, if the method prescribes using deuterated internal standards or matrix-matched calibration standards, then the reported analyte recoveries should be calculated according to those procedures.

SRM: Standard Reference Material. A certified reference material issued by the National Institutes of Standards and Technology (NIST) in the United States. (www.nist.gov/SRM).

Standard: A substance of known identity and purity and/or concentration.

Systematic error: Component of measurement error that in replicate measurements remains constant or varies in a predictable manner. This may also be referred to as *Bias*.

Threshold Value: In qualitative analysis, the concentration of the analyte that is either statistically lower than the level of concern (for limit tests) or at which positive identification ceases (for confirmation of identity methods). See also *Cut-off Concentration*.

Trueness: The degree of agreement of the mean value from a series of measurements with the true value or accepted reference value. This is related to systematic error (bias).

Uncertainty: Non-negative parameter characterizing the dispersion of the values being attributed to the measured value. See 3.3

UAP: Unfortified Analytical Portion.