U.S. Department of Health & Human Services

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Elemental Analysis Manual

for Food and Related Products

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Glossary and Acronyms

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Below is a non-inclusive list of technical terms and acronyms used in the EAM. Chemical names are omitted (e.g., *monomethylarsonic acid* and its acronym MMA) as well as common terms (e.g., *pipettes*, *solutions*) and terms used in only specific applications (e.g., in method 4.10 *ready to drink* and its acronym RTD).

GLOSSARY

- **Accuracy:** The closeness of agreement between a test result and an accepted reference value. (Discussed in EAM 3.2.1. A related term is *trueness*.) 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.

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

- **Analytical Solution Detection Limit (ASDL):** The lowest level that can be detected in a test solution obtained after a test portion is digested. See EAM 3.2.1

- **Analytical Solution Quantification Limit (ASQL):** The level in a test solution which is obtained after a test portion is digested and that corresponds with the defined quantitation specification. It converts to LOQ. See also EAM 3.2.1

- **Batch:** The combination of analytical portions, standards, blanks, and all control materials 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. (Discussed in EAM 3.2.2).

- **Bias:** The difference between the expectation of the test result and the actual, or true, value. In application, the accepted reference value is taken to be the true value. Bias causes results to have systematic error. There may be multiple components contributing to the total bias.

- **Blank:** A generic term that can refer to anything that contributes, or could contribute, analyte in addition to that which is intrinsically present within a test portion. Blank level refers to the amount of analyte that would give bias if not subtracted from a measurement result. Blank materials or items can be classified as, e.g., method blanks, matrix blanks, reagent blanks, instrument blanks, and field blanks. Blanks are differentiated from contamination in that they occur normally and consistently during the chemical measurement process whereas contamination varies and is not a part of normal operations. While both of these are unwanted, blank is predictable and can be subtracted but contamination is problematic. Blanks are addressed in the methods and EAM 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 analytical portion measured. Typically, this is accomplished through the use of calibration standards containing known analyte levels.

- **Calibration Standard:** A substance (usually a solution) with a well-known level (mass fraction, concentration, etc.) of analyte used to calibrate the measuring/detection system. May be matrix matched for specific sample matrices.

- **Carryover:** Residual analyte from a previous analytical portion (or standard) which is retained in the analytical system and measured in subsequent analyses. Carryover is also commonly expressed as “memory” or “memory effect”.

- **Certified Reference Material (CRM):** A very carefully prepared material that has known analyte levels and is accompanied by documentation (i.e., a certificate) issued by an authoritative body and which provides one or more specified property values with associated uncertainties and traceability, using valid procedures. (Discussed in EAM 3.5. 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

- **Characteristic Mass (m_o):** 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

- **Check Analysis:** A second independent analysis the result of which is compared with that from the initial analysis. Check analyses are typically performed by a different analyst using the same method.

- C**heck solution (CS):** Solution with analytes at known concentrations that is analyzed periodically during and at the end of an analytical run. (Discussed in EAM 3.2.4)

- **Cold Vapor-Atomic Absorption Spectrometry (CVAAS):** See, for example, Method 4.5

- **Dilution Factor (DF):** 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. (Discussed in EAM 3.4.3)

- **Error:** The difference between a measured value and the true value. Two examples of how error is expressed in common analytical situations are 1) as a recovery (the difference between a measurement result and a certified value or a spike level) and 2) as a standard deviation (a statistical evaluation of the distribution of a series of results about the mean). A related term is *uncertainty* - the range (about a measurement) within which the true value is believed to lie. Whereas error is an *a posteriori knowledge* (i.e., after-the-fact finding of what has been observed), uncertainty is an *a priori estimate* (i.e., is a before-the-fact prediction).

- **False Negative:** Failure to decide a substance is present when it is. Also called error of the second kind.

- **False Positive:** Deciding a substance is present when it is not. Also called error of the first kind.

- **Fortification Recovery:** See Percent Difference.

- **Fortified Analytical Portion (FAP):** Analytical portion that was fortified (spiked) with analyte before digestion. It is used to determine if the preparation procedure or sample matrix contribute bias to the analytical result. (Discussed in EAM 3.2.4)

- **Fortified Analytical Solution (FAS):** Analytical solution that is fortified (spiked) with analyte(s) before instrumental determination of analyte concentration. It is used to determine the need for further dilution of the analytical solution to account for matrix effects. (Discussed in EAM 3.2.4)

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

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

- **Independent Check Solution (ICS):** 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. Used to confirm instrument stability. (Discussed in EAM 3.2.4)

- **Inductively Coupled Plasma-Mass Spectrometry (ICP-MS):** See, for example, Methods 4.7, 4.8, 4.10, and 4.11

- **Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES):** Formerly referred to as inductively coupled plasma-atomic emission spectrometry (ICP-AES). See, for example, Methods 4.4 and 4.6

- **Instrumental Detection Limit (IDL):** The lowest level that an instrument's detector can measure. It represents an ideal case (e.g., without matrix effect). The terms *limit of detection* and *analytical solution detection limit* apply to a real-world samples.

- **Interference:** An effect (positive or negative) on analyte measurements. It may be produced by a substance other than the analyte and/or include spectral, physical, or chemical properties. Interferences bias results. In extremes, interference can prevent an analysis.

- **Internal Standard (ISTD):** A chemical added to a test portion, in known quantity and at a specified stage in the analysis. It facilitates quantitation of the analyte and is used to compensate for matrix effects, incomplete spike recoveries, etc. Analyte mass fraction (or concentration) is deduced from its response relative to that produced by the internal standard. Internal standards should have similar physico-chemical properties to those of the analyte.

- **Laboratory method blank (MBK_L):** The laboratory's running average MBK level. (Discussed in EAM 3.2.1)

- **Limit of Detection (LOD):** The minimum level (mass fraction, concentration, amount, etc.) 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 MDL (Method Detection Limit). See EAM 3.2.1

- **Limit of Quantitation (LOQ):** The minimum level (mass fraction, concentration, amount, etc.) 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 also EAM 3.2.1

- **Linear Dynamic Range (LDR):** The linear portion of a response curve.

- **Linearity:** The degree to which instrument response is evenly proportional to analyte level. It relates to the section of instrument response curve (standard curve) that is linear (or nearly linear).

- **Mass Correction Factor (MCF):** Factor applied to analytical portion mass to account for dilution. This would apply, for example, if water (or other solvent) is added to aid homogenization of analytical portion or if an analytical solution is diluted to reduce matrix effects. (Given in EAM 3.4.6)

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

- **Matrix Blank:** In usage, can refer to a matrix blank level or matrix blank material (or portion of material). It involves a substance that closely matches the sample material being analyzed with regard to matrix components. Ideally, the matrix blank has very similar composition but does not contain the analyte(s) of interest. It 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:** The influence that the matrix has on analyte measurement. The term can refer to a process (e.g., "The matrix depresses the signal.") or a numerical change (e.g., "The matrix enhances the signal by 10%.").

- **Method blank (MBK):** MBK usually refers to a MBK portion (e.g., "MBK #1 is in the autosampler's position 15"). It can also, however, refer to a MBK level (e.g., "The typical MBK is about 5 ng/kg"). No distinction is made between these two concepts because the context is always obvious. MBKs may consist of matrix material but are often aliquots of reagent water. They are subjected to all sample processing operations including all reagents used to analyze the analytical portions. The analyte(s) of interest will ideally be absent or detectable at levels that are extremely consistent. (Discussed in EAM 3.2.1)

- **Method blank critical level (MBK_C):** The laboratory's MBK critical value level. (Discussed in EAM 3.2.1)

- **Method Detection Limit:** Term commonly used in analytical activities associated with EPA programs. EPA defines it as the "... minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results." Importantly, the basis for MDL is that it applies to only whether something is observed (i.e., addresses only false positives). It does not account for whether something is missed (i.e., does not address false negatives). Thus, it shows how high a signal needs to be to say it is "detected" but not high enough to say an analyte won't be missed. In practice, MDL is usually set at 99% confidence (false positives only) and LOD at 95% confidence (for both false positives and false negatives). Although LOD is greater than MDL, the numerical difference between them is therefore not great. (LOD is emphasized in EAM methods.)

- **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, and ruggedness.

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

- **Percent Difference (PD):** Compares a measured value with a reference (or true) value and is an expression of accuracy. It is equal to the difference between the two numbers divided by the true value and is expressed in percent. In the context of fortification (spiking), it may be called percent recovery, fortification recovery, or spike recovery. Whereas PD is an accuracy term, relative percent difference (a similar but different calculation) is a precision term. (EAM 3.4.5)

- **Percent Recovery:** See Percent Difference.

- **Precision:** The closeness of agreement between independent test results obtained under specified conditions. It is described by statistical methods such as a standard deviation or relative percent difference. See also *Random Error*. Precision can be further classified as *Repeatability* and *Reproducibility*. See 3.2.1

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

- **Reagent Blank:** Reagent blank can be an item (reagents used in the procedure taken through the entire method) or a level (e.g., "reagent blank was found to be <0.2 ng/kg"). 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) that remains after an analytical procedure. For example, it may be recovery of an element after a CRM is analyzed or fortification recovery. Recovery is usually expressed as a percentage (i.e., percent recovery) but could also be in absolute terms. See also Percent Difference.

- **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 standards traceable to a standards body such as the National Institute of Standards and Technology (NIST).

- **Relative Percent Difference (RPD):** Compares measured values with each other and is an expression of precision that is generally used when only two measurement results are available (standard deviation is used when there are n>2 replicates). It is equal to the difference between the two numbers divided by the mean of the two and is expressed in percent. Whereas RPD is a precision term, percent difference is an accuracy term.

- **Repeatability:** Precision obtained for replicate measurements made under identical or similar conditions (e.g., one method, replicates of one homogeneous material, in the same test facility, by one analyst, using the same equipment, and within a short interval of time.) See 3.2.1

- **Reproducibility:** Precision obtained for replicate measurements made using one method but under varying conditions, such as in different test facilities, with different operators, using different equipment, possibly over an extended period of time, etc. See 3.2.1

- **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. Although distinction between the two terms is sometimes made in scientific literature, their meanings are so closely related that, for all practical purposes, the two are used interchangeably. Since multiple authors contribute to the EAM, both will appear.

- **Sensitivity:** Two meanings. In a metrological context where the focus is on quantitation, sensitivity is the change in instrument response which corresponds to a change in the measured quantity (i.e., instrument calibration slope). One instrument would be more sensitive than another if it has a greater change in signal response with small changes in analyte solution mass fraction. In a qualitative context where the focus is on detection, sensitivity is synonymous to limit of detection. One method would be more sensitive than another if it can detect a contaminant at lower levels.

- **Spike Recovery:** See Percent Difference.

- **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*.

- **Trueness:** The degree of agreement of the mean value from a series of measurements with the true or accepted reference value. This is related to systematic error (bias). Discussed in EAM 3.2. A related term is accuracy.

- **Uncertainty:** The range (about a measurement) within which the true value is believed to lie and is relative to some level of confidence (usually 95%). A related term is *error* - the difference between a measured value and the true value. Whereas uncertainty is an *a priori estimate* (i.e., is a before-the-fact prediction), error is an *a posteriori knowledge* (i.e., after-the-fact finding of what has been observed). (Discussed in EAM 3.3)

ACRONYMS

- **ASDL:** Analytical Solution Detection Limit.

- **ASQL:** Analytical Solution Quantification Limit.

- **CRM:** Certified Reference Material
- **CS:** Check solution.
- **CVAAS:** Cold Vapor-Atomic Absorption Spectrometry.
- **DF:** Dilution Factor.
- **FAP:** Fortified Analytical Portion.
- **FAS:** Fortified Analytical Solution.
- **FMB:** Fortified Method Blank.
- **GFAAS:** Graphite Furnace-Atomic Absorption Spectrometry.
- **HPLC:** High Performance Liquid Chromatography.
- **ICP-MS:** Inductively Coupled Plasma-Mass Spectrometry.
- **ICP-OES:** Inductively Coupled Plasma-Optical Emission Spectrometry.
- **ICS:** Independent Check Solution.
- **IDL:** Instrumental Detection Limit.
- **ISTD:** Internal Standard.
- **LDR:** Linear Dynamic Range.
- **LOD:** Limit of Detection.
- **LOQ:** Limit of Quantitation.
- **MBK:** Method Blank.
- **MBK_C:** Method blank critical level.
- **MBK_L:** Laboratory method blank.
- **MCF:** Mass Correction Factor.
- **MDL:** Method Detection Limit.
- **m_o :** Characteristic Mass.
- **PD:** Percent Difference.

- **RM:** Reference material.
- **RPD:** Relative Percent Difference.
- **SRM:** Standard Reference Material.
- **UAP:** Unfortified Analytical Portion.