



Standard Practice for 99 %/95 % Interlaboratory Detection Estimate (IDE) for Analytical Methods with Negligible Calibration Error¹

This standard is issued under the fixed designation D6091; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice establishes a standard for computing a 99 %/95 % Interlaboratory Detection Estimate (IDE) and provides guidance concerning the appropriate use and application. The calculations involved in this practice can be performed with DQCALC, Microsoft Excel-based software available from ASTM.²

1.2 The IDE is computed to be the lowest concentration at which there is 90 % confidence that a single measurement from a laboratory selected from the population of qualified laboratories represented in an interlaboratory study will have a true detection probability of at least 95 % and a true nondetection probability of at least 99 % (when measuring a blank sample).

1.3 The fundamental assumption of the collaborative study is that the media tested, the concentrations tested, and the protocol followed in the study provide a representative and fair evaluation of the scope and applicability of the test method as written. When properly applied, the IDE procedure ensures that the 99 %/95 % IDE has the following properties:

1.3.1 *Routinely Achievable IDE Value*—Most laboratories are able to attain the IDE detection performance in routine analyses, using a standard measurement system, at reasonable cost. This property is needed for a detection limit to be practically feasible. Representative laboratories must be included in the data to calculate the IDE.

1.3.2 *Routine Sources of Error Accounted For*—The IDE should realistically include sources of bias and variation which are common to the measurement process. These sources include, but are not limited to: intrinsic instrument noise, some typical amount of carryover error, plus differences in laboratories, analysts, sample preparation, and instruments.

1.3.3 *Avoidable Sources of Error Excluded*—The IDE should realistically exclude avoidable sources of bias and variation, that is, those which can reasonably be avoided in

routine field measurements. Avoidable sources would include, but are not limited to: modifications to the sample, measurement procedure, or measurement equipment of the validated method, and gross and easily discernible transcription errors (provided there was a way to detect and either correct or eliminate them).

1.3.4 *Low Probability of False Detection*—The IDE is a true concentration consistent with a measured concentration threshold (critical measured value) that will provide a high probability, 99 %, of true nondetection (a low probability of false detection, $\alpha = 1$ %). Thus, when measuring a blank sample, the probability of not detecting the analyte would be 99 %. To be useful, this must be demonstrated for the particular matrix being used, and not just for reagent water.

1.3.5 *Low Probability of False Nondetection*—The IDE should be a true concentration at which there is a high probability, at least 95 %, of true detection (a low probability of false nondetection, $\beta = 5$ %, at the IDE), with a simultaneous low probability of false detection (see 1.3.4). Thus, when measuring a sample at the IDE, the probability of detection would be at least 95 %. To be useful, this must be demonstrated for the particular matrix being used, and not just for reagent water.

NOTE 1—The referenced probabilities, α and β , are key parameters for risk-based assessment of a detection limit.

1.4 The IDE applies to measurement methods for which calibration error is minor relative to other sources, such as when the dominant source of variation is one of the following (with comment):

1.4.1 *Sample Preparation*, and calibration standards do not have to go through sample preparation.

1.4.2 *Differences in Analysts*, and analysts have little opportunity to affect calibration results (such as with automated calibration).

1.4.3 *Differences in Laboratories*, for whatever reasons, perhaps difficult to identify and eliminate.

1.4.4 *Differences in Instruments* (measurement equipment), which could take the form of differences in manufacturer, model, hardware, electronics, sampling rate, chemical processing rate, integration time, software algorithms, internal signal processing and thresholds, effective sample volume, and contamination level.

¹ This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.02 on Quality Systems, Specification, and Statistics.

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² Available from ASTM International Headquarters. Order Adjunct No. ADJDQ-CALC. Original adjunct produced in 2007.

1.5 *Alternative Data Quality Objectives*—Other values for α , β , confidence, etc. may be chosen for calculating an IDE; however, this procedure addresses only the 99 %/95 % IDE.

2. Referenced Documents

2.1 *ASTM Standards*:³

D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

2.2 *ASTM Adjuncts*:

DQCALC Microsoft Excel-based software for the Interlaboratory Quantitation Estimate (IQE)²

3. Terminology

3.1 *Definitions*:

3.1.1 99 %/95 % *Interlaboratory Detection Estimate* (99 %/95 % IDE, also denoted LD for *Limit of Detection* in accordance with Currie (1)⁴—The lowest concentration at which there is 90 % confidence that a single measurement from a laboratory selected from the population of qualified laboratories represented in an interlaboratory study will have a true detection probability of at least 95 % and a true nondetection probability of at least 99 %.

3.2 *Definitions of Terms Specific to This Standard*:

3.2.1 *Censored Measurement*—A measurement that is not reported numerically nor is reported missing but as a nondetect or a less-than, for example, “less than 0.1 ppb.” The former means that an algorithm in the measurement system determined that the measurement should not be reported numerically for one of two reasons: either it was considered not sufficiently precise or accurate, or the identification of the analyte was suspect. A reported less-than may have the same meaning, but it also implies (perhaps erroneously) that any concentration greater than or equal to the accompanying value (for example, 0.1 ppb) can be measured and will be reported numerically.

3.2.2 *Detection Limit (DL) or Limit of Detection (LD)*—A numerical value, expressed in physical units or proportion, intended to represent the lowest level of reliable detection (a level which can be discriminated from zero with high probability while simultaneously allowing high probability of nondetection when blank samples are measured.

NOTE 2—In some cases, the discrimination may be from a value other than zero, such as a background level. Note also that a DL also depends on other characteristics of the measurement and detection process, such as described in 1.3.2. The IDE is an example of a DL.

3.2.3 *Probability of False Detection*—The false positive probability, denoted α , that a single measurement of a blank sample will result in a detection. (See Fig. 1.) This probability is often referred to as the Type 1 error probability and depends on the analyte, measurement system, analytical method, matrix, analyst, and measurement (recovery) threshold (measurement critical value) used to decide whether detection has occurred. This definition can be generalized to refer to unwanted detection from a single measurement of a sample at any nonzero concentration of the analyte rather than a blank sample, provided that the nonzero concentration is less than the detection limit or IDE.

3.2.4 *Probability of False Nondetection*—The false negative probability, denoted β or $\beta(T)$, that a single measurement of a sample containing a nonzero concentration, T , of an analyte of interest will result in a nondetection. This is the complement of the probability of true detection. (See Fig. 1.) This probability function is often referred to as the Type 2 error probability function, and it depends explicitly on the concentration (T). It depends implicitly on the analyte, measurement system, analytical method, matrix, analyst, and critical value for detection.

3.2.5 *Probability of True Detection*—The probability, denoted $1-\beta$ or $1-\beta(T)$, that a single measurement of a sample containing a nonzero concentration, T , of an analyte of interest will result in a detection. (See Fig. 1.) This probability is often referred to as statistical power or the power of detection, and it depends explicitly on the concentration (T). It depends implicitly on the analyte, measurement system, analytical method, matrix, analyst, and critical value for detection.

3.2.6 *Probability of True Nondetection*—The true negative probability, denoted $1-\alpha$, that a single measurement of a blank

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

⁴ The boldface numbers in parentheses refer to the list of references at the end of this standard.

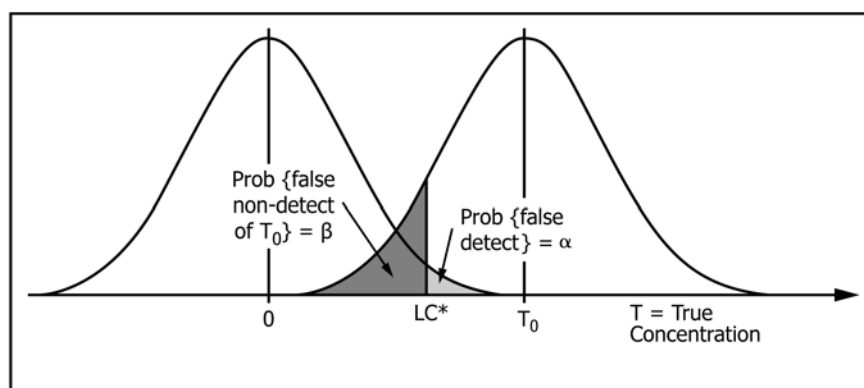


FIG. 1 Simplest Case of Reliable Detection