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Standard Guide for Reporting Results of Analysis of Water[1]

This standard is issued under the fixed designation D 596; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This guide provides guidelines for reporting inorganic and organic results of analyses of drinking water, waste water, process water, ground water, and surface water, and so forth, to laboratory clients in a complete and systematic fashion.

1.2 The reporting of bacterial and radiological data are not addressed in this guide.

1.3 The commonly used data qualifiers for reviewing and reporting information are listed and defined. Client and laboratory specific requirements may make use of other qualifiers. This guide does not preclude the use of other data qualifiers.

1.4 This guide discusses procedures for and specific problems in the reporting of low level data, potential errors (Type I and Type II), and reporting data that are below the calculated method detection limit and above the analyte.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 933 Practice for Reporting Results of Examination and Analysis of Water-Formed Deposits

D 1129 Terminology Relating to Water

D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water

D 3856 Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water

D 4210 Practice for Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data[3]

D 4460 Practice for Calculating Precision Limits Where Values are Calculated from Other Test Methods

D 4840 Guide for Sample Chain-of-Custody Procedures

D 5792 Practice for Generation of Environmental Data Related to Waste Management Activities: Development of Data Quality Objectives

D 6091 Practice for 99 %/95 % Interlaboratory Detection Estimate (IDE) for Analytical Methods with Negligible Calibration Error

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

3. Terminology

3.1 Definitions—For definitions of terms used in this practice, refer to Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *surrogates*—compounds that are similar to analytes of interest in chemical composition and behavior, separation, and measurements, but that are not normally found in environmental samples.

NOTE 1—These compounds are added to blanks, standards, samples, or spiked samples prior to analysis to confirm the proper operation of the analytical system.

3.2.2 *Type I error*—a statement that a substance is present when it is not.

3.2.3 *Type II error*—a statement that a substance was not present (was not found) when the substance was present.

4. Significance and Use

4.1 The proper use of analytical data requires adequate documentation of all inputs, that is, the source and history of the sample, laboratory performing the analysis, method of analysis, date of analysis, precision and bias of the measurements, and related quality assurance information.

4.2 In order to have defensible data, the report must be complete and accurate, providing adequate information to evaluate the quality of the data and contain supporting information that documents sampling and analysis procedures.

4.3 This guide contains some of the common data qualifiers or "flags" commonly used by laboratories following the Good Laboratory Practices, the Government Contract program, or found in the commercial laboratories. Examples of these qualifiers are the use of (E) for estimated value, (U) for analyzed for but not detected, and (B) for analyte was found in the blank (see 8.11). The qualifiers included in this guide should help the laboratory and its customers to better understand each other by using standardized qualifiers.

[1] This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.02 on General Specifications, Technical Resources, and Statistical Methods.

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[2] *Annual Book of ASTM Standards*, Vol 11.02.

[3] Withdrawn.

4.4 Practice D 933 is a comprehensive practice for reporting water-formed constituents such as metal oxides, acid anhydrides, and others.

5. Sample Documentation

5.1 Information regarding the source and history of the sample to be included in the analytical report should define the sample and include the following, as appropriate:

5.1.1 Laboratory performing analysis,

5.1.2 Name and address of organization or person requesting analysis,

5.1.3 Specific location of sampling and complete identification of sample,

5.1.4 Date and time of sampling,

5.1.5 Sample identification number, and

5.1.6 Sampling method, treatment, and preservation.

5.2 In addition to the information in 5.1, the following information should be included as appropriate:

5.2.1 Identification of sampling organization and individual sampler,

5.2.2 Pressure and temperature of system sampled,

5.2.3 Flow rate of water in a stream, outfall, pipe, and so forth.

5.2.4 Copies of sampling logs with signatures,

5.2.5 Chain-of-custody forms with signatures (see Guide D 4840),

5.2.6 Results of field measurements, and

5.2.7 Description information (color, odor, and so forth) clearly presented.

5.2.8 The information about the sample documented in the report should be complete enough to provide direct unabridged links to underlying documents (such as chain-of-custody records and field logs) and information (such as name of sampler, lot numbers of the sample bottles, and preservatives).

6. Analysis Documentation

6.1 The laboratory system shall provide enough information to the user or reviewer so that all of the events that could influence the quality of the data can be reconstructed. The user may not need to have the information communicated directly to them, but it must be available upon request. Such information should describe how effectively all procedures were carried out and how processes were controlled so that they meet industry and government standards for performance.

6.2 As described in Guide D 3856, the test method of analysis should be specified in the analytical report for each determination performed on a sample. A reference of sufficient definition or a copy of the test method should be provided to the requestor of the analytical services.

6.3 The report should note any deviation from the specified test method. Whenever a choice is allowed, the rational for selecting a given method should be documented.

6.4 The precision, bias, and detection limit of each analytical test method should be disclosed as part of either the test method or the analytical report. Consult Guide D 3856 for the quality control system from which estimates of precision and bias could be made, or review the procedure for determining single-operator precision of a test method as provided in Practice D 2777 for guidance. The procedure used to derive the detection limit should be identified along with any specific definitions associated with the derivation. Practice D 4210 is one of many sources for computing single laboratory method detection limits. Practice D 6091 provides an estimate of the detection level achievable by multiple laboratories on single sample.

6.5 The date and time on which each determination is performed should be recorded, as should other time-critical processes such as extractions, storage times, drying times, and so forth.

6.6 The analytical reports should clearly specify the form in which multi-atomic analytes, such as nitrate and orthophosphate, are reported.

6.7 If a sample is prepared for analysis in a nonstandard manner or in a manner different from the routine batch procedures (that is, special cleanup procedures or dilution required prior to analysis) then the report should clearly present the deviation and the reason why the deviation was required.

6.8 If a sample is diluted prior to analysis, the sample dilution values should be reported from which the ratios can be determined and the reason for the dilution documented.

7. Documentation of Quality

7.1 Each sample analysis may have different quality needs based on the use of the data or the Data Quality Objectives (See Practice D 5792). This information should be determined before sampling and analysis. Based on the information, an analytical report may include the following information, as appropriate:

7.1.1 Amount recovered and percent recovery of any surrogate compounds with laboratory control limits,

7.1.2 Results of corresponding check samples or blank spikes with laboratory control limits,

7.1.3 Results of analysis of duplicate samples or duplicate matrix spike samples and the percent difference with laboratory control limits,

7.1.4 Recoveries of any matrix spikes (and matrix spike duplicates) with laboratory control limits,

7.1.5 Results of all blanks,

7.1.6 Results of any reference samples run during sample analysis with laboratory control limits,

7.1.7 Calibration and tuning data, and

7.1.8 Chromatogram or charts.

8. Reporting Data

8.1 Report data in accordance with the customer and laboratory agreement. In the absence of a specific agreement, report the data in accordance with laboratory policy or government mandated requirements, if appropriate.

8.2 Compound specific analysis may require tentative identification without verification. The criteria for identification and a copy of the chromatogram or other instrument output should be included in the report.

8.3 Upon request, the quality documentation found in Section 7 should be included in the report.

8.4 Any deviation from the established method or standard operating procedure (SOP), must be reported to the customer. Reasons for the deviation and the expected impact on the data should be given.

8.5 The procedures, method, or SOP used to report the analytical values shall be specified.

NOTE 2—If there is no deviation from the contract or agreed upon procedure, then reference to the document may be sufficient.

8.6 In cases where the customer desires a summary of the data to be transmitted to them, the laboratory will keep sufficient records on file to reproduce the data.

8.7 Detection limits should be reported in accordance with laboratory policy, established procedures, or regulatory requirement. These polices and procedures must be clearly identified and understood by all personnel reporting the analysis. Results reported below laboratory established detection limits may be reported upon customer request as discussed in Section 10.

NOTE 3—Some commercial laboratories establish their detection limits based on what their average laboratory can achieve over an extended period of time. A given laboratory may achieve lower compound specific values than the average.

8.8 Report blank data results and, where appropriate, actual data from the equipment. Blanks should not be subtracted from the sample results unless required by the test method. The customer should determine, with advisement form the laboratory, if blank subtraction is necessary or required. (See Section 10).

8.9 Recording direct measurement test results should be reported by recording all digits that are known plus one that may be subject to change on repeated analysis. When calculating results from test data, rounding should be performed only on the final result, not upon the intermediate values employed in the calculation.

8.10 Frequently, replicate determinations are made. When replicate results are obtained, useful information is now available that is lost if the results of these replicates are not reported. It is important that a reporting laboratory establish a consistent protocol for reporting replicate data. In order to arrive at a coherent protocol for this purpose, a number of issues and options should be evaluated.

8.10.1 *Replicate Types*—Replication may be performed at different levels. Replication may occur at the point of sampling, at the sample preparation step, the prepared sample analysis step, or at some other point in the analytical process. Different types of replicates may be handled differently and should not be mixed. The type of replicate should be made clear to the user.

8.10.2 *Reporting Replicate Averages*—Replicate results may be reported separately or as an average. When average results are reported, several factors are considered.

8.10.2.1 *Documentation*—The data users should know when the reported results is an average of replicates. Averages of different numbers or replicates have different quality (precision) leading to different conclusions about data validity. For this reason, the number of replicates used in a reported average should be reported with the averaged results.

8.10.2.2 *Criteria*—Criteria must be established as to when a result is part of a replicate set. For example, when a dilution is performed on a sample prior to analysis, the original result and the diluted result may both be within the quantitative range of the analytical method. Although the dilution step produces a value that is not a true replicate, the added value of reporting an average of these values may be warranted.

8.10.2.3 *Selection for Averaging*—Analytical results may be produced within four discrete ranges. Each of these ranges is affected by sample dilution or concentration. Replicates may be generated within different ranges for the sample analysis. The four discrete ranges are listed as follows in increasing order of size:

(*1*) Below a limit of detection, where the analyte cannot be said to be present with confidence above a set level.

(*2*) Between a limit of detection and a limit of quantitation where the analyte can be said to be present with a preset limit of confidence but the concentration value does not meet a preset criteria.

(*3*) Between a limit of quantitation and the upper limit of the quantitation range of the analytical method. This is the quantitation range of the analytical method. This is typically the highest calibration standard used.

(*4*) Above the quantitation range of the analytical method.

8.10.2.4 It is important to first establish which of the ranges found in 8.10.2 is applicable to each replicate. Replicates should not be averaged across ranges. The following selection criteria for averaging should be followed:

(*1*) Select and average only replicates that fall within the quantitation range of the analytical method. If none exist, then,

(*2*) Select and average only replicates that fall above the quantitation range of the analytical method. If none exist, then

(*3*) Select and average only replicates that fall between a limit of detection and a limit of quantitation. If none exist, then

(*4*) Select and average only replicates that fall above the established limit of detection.

NOTE 4—References to range refer to ranges adjusted for sample concentration or dilution.

8.10.2.5 *Exclusion of Data*—Individual values may be excluded from an average for other data quality reasons.

8.11 All data should be reported with an appropriate number of significant figures. Significant figures represent the precision or the degree of quantitative uncertainty in the result. Too many figures in a result indicate a smaller relative standard deviation in the measurement than is warranted. The usual convention for significant figure reporting is to retain one uncertain figure.

8.11.1 There is a direct relationship between relative standard deviation and the number of significant figures, that is, the number of significant figures is an inverse function of the relative standard deviation (RSD).

8.11.1.1 Since most measurement systems demonstrate an increasing RSD with decreasing concentration, the number of significant figures decreases as the concentration decreases. At approximately the quantitation limit, there should be only one significant figure. Data at the approximate quantitation limit becomes uncertain. By extension, at the detection limit, there are no significant figures making quantitation impossible since there is no confidence in the presence of the measured analyte.

8.11.1.2 The quantitation limit chosen, that is, the point where there is one significant figure, is a function of the lowest acceptable or achievable RSD. With each decade of measured concentration increase and associated RSD decrease, one