



Standard Guide for Reporting Results of Analysis of Water¹

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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:

D 933 Practice for Reporting Results of Examination and Analysis of Water-Formed Deposits²

D 1129 Terminology Relating to Water³

D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water³

D 3856 Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water³

D 4210 Practice for Interlaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data³

D 4460 Practice for Calculation of Precision Limits Where Values are Calculated from Other Test Methods⁴

D 4840 Guide for Sampling Chain-of-Custody Procedures³

D 5792 Practice for Generation of Environmental Data Related to Waste Management Activities: Development of Data Quality Objectives⁵

D 6091 Practice for 99 %/95 % Interlaboratory Detection

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 *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Annual Book of ASTM Standards*, Vol 04.03.

⁵ *Annual Book of ASTM Standards*, Vol 11.04.

Estimate (IDE) for Analytical Methods with Negligible Calibration Error³

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications⁶

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.

⁶ *Annual Book of ASTM Standards*, Vol 14.02.

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 rationale 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 policies 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 from 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

TABLE 1 Data Qualifiers

U—The element or compound was measured, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
J—The associated value is an estimated quantity.
R—The data are unusable. The reason should be specified for the data being unusable.
Note—Analyte may or may not be present.
E—The reported value is estimated because of the presence of interference.
M—Duplicate injection precision is not met.
N—Spiked sample recovery is not within control limits.

additional significant figure can be added until the RSD levels off. At which point, the maximum number of significant figures is reached.

8.11.2 Practice E 29 is a worthwhile document to review for a discussion of the principles and practices for determining significant figures.

8.12 When a value is computed from two or more other test results, refer to Practice D 4460 for techniques of determining precision limits of the calculated value.

9. Review of Analytical Results

9.1 All data should have a peer review before being finalized. A further review should be done by the project leader or equivalent to ensure the customer's requirements have been met.

9.2 At some preselected frequency, electronic data should be hand-calculated to verify proper operation. This check should be documented and kept with data files.

9.3 The procedures and codes used to report analytical values should be consistent with those found in Table 1.

9.3.1 Qualifiers are used by the analyst, data reviewers, and government agencies in the contract laboratory program to describe and qualify data. They are an effective form using letters to explain a reported value, that is, methylene chloride 5 ppb, J., where the analyte concentration was estimated (J) to be 5 ppb in the sample. Recommended qualifiers are listed as follows. A complete list may be found in the Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analysis⁷ and Office of Solid Waste and Emergency Response Laboratory Data Validation Functional Guidelines for Evaluating Organic Analysis⁸.

9.4 Cations and anions balance may be used to determine how logical the results are. Table 2 lists factors for interconversion between units in common use and Table 3 list factors for interconversion of milligrams per litre (mg/L) and milliequivalent per litre (meq/L) of common ions.

9.4.1 The deviation from a perfect balance between cations and anions determined in water samples may be appraised by totalling separately the determined concentrations in milliequivalent per litre of anions and cations. This can only be

TABLE 2 Conversion Factors Between Units in the Specific Test Method and Other Units in Common Use

To Convert	To	Multiply By
mL (or cm ³) of dissolved oxygen/L	mg/L	1.429
grains/US gal	mg/L	17.12
grains/Imperial gal	mg/L	14.25
grams/L	mg/L	1000
normality	meq/L	1000
mg/L as CaCO ₃	meq/L	0.0200
grains/US gal as CaCO ₃	meq/L	0.342
grains/Imperial gal as CaCO ₃	meq/L	0.285
mg of dissolved oxygen per L	mL (or cm ³) of dissolved oxygen per L	0.700
mg/L	grains/US gal	0.0584
mg/L	grains/Imperial gal	0.0702
mg/L	grams/L	0.001
meq/L	normality	0.001
meq/L	percent of normal	0.1
meq/L	mg/L as CaCO ₃	50.0
meq/L	grains/US gal as CaCO ₃	2.92
meq/L	grains/Imperial gal as CaCO ₃	3.51

TABLE 3 Factors for Interconversion Between Milligrams Per Litre and Milliequivalents Per Litre^{A,B}

Ion	Multiplier	
	mg/L to meq/L	meq/L to mg/L
Al ⁺³	0.1112	8.994
Ba ⁺²	0.01456	68.67
Br ⁻	0.01252	79.90
Ca ⁺²	0.04990	20.04
Cl ⁻	0.02821	35.45
CN ⁻	0.03844	26.02
CO ₃ ⁻²	0.03333	30.00
Cr ⁺³	0.05770	17.33
Cr ⁺⁶	0.1154	8.666
CrO ₄ ⁻²	0.01724	58.00
Cu ⁺²	0.03147	31.77
F ⁻	0.05264	19.00
Fe ⁺²	0.03581	27.92
Fe ⁺³	0.05372	18.62
H ⁺	0.9921	1.008
HCO ₃ ⁻	0.01639	61.02
HPO ₄ ⁻²	0.02084	47.99
H ₂ PO ₄ ⁻	0.01031	96.99
HS ⁻	0.03024	33.07
HSO ₃ ⁻	0.01233	81.07
HSO ₄ ⁻	0.01030	97.07
I ⁻	0.007880	126.9
K ⁺	0.02558	39.10
Mg ⁺²	0.08229	12.15
Mn ⁺²	0.03640	27.47
Mn ⁺⁴	0.07281	13.73
Na ⁺	0.04350	22.99
NH ₄ ⁺	0.05544	18.04
Ni ⁺²	0.03407	29.35
NO ₂ ⁻	0.02174	46.01
NO ₃ ⁻	0.01613	62.00
OH ⁻	0.05880	17.01
Pb ⁺²	0.009653	103.6
PO ₄ ⁻³	0.03159	31.66
S ⁻²	0.06238	16.03
SiO ₃ ⁻²	0.02629	38.04
SO ₃ ⁻²	0.02498	40.03
SO ₄ ⁻²	0.02082	48.03
Sr ⁺²	0.02283	43.81
Zn ⁺²	0.03059	32.69

^A Based on ¹²C = 12 amu (atomic mass units).

^B It is assumed that reactions proceed to the zero oxidation state.

⁷ Office of Solid Waste and Emergency Response Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analysis, Pub. 9240.120, December 1994

⁸ Office of Solid Waste and Emergency Response Laboratory Data Validation Functional Guidelines for Evaluating Organic Analysis, PUB. 9240.1-27, December 1994

done if all major ions have been determined. According to Friedman and Erdmann in their chapter titled "Quality Assurance Practices for Chemical and Biological Analyses of Water

and Fluvial Sediments”⁹, the cation-anion difference, either positive or negative, may be calculated from the following empirical formula in which cations and anions are expressed in milliequivalent per litre.

$$\text{percent cation - anion difference} = \frac{\epsilon \text{ cations} - \epsilon \text{ anions}}{\epsilon \text{ cations} + \epsilon \text{ anions}} \times 100$$

NOTE 5—A study by J.D. Hem titled “Study and Interpretation of the Chemical Characteristics of Natural Water”¹⁰ states with careful work, the difference will not generally exceed 2 % of the total cations or anions in waters of moderate concentrations (250 to 1000 mg/L). A somewhat larger percentage can be tolerated if the sum of cations and anions is less than about 5.00 meq/L.

9.4.2 In addition to the cation balance, other types of analytical data can be used to test for logical consistency. These kinds of tests have the general form of testing for the whole being equal to or less than the sum of its parts. These tests can be done within analysis, between analyses, and between samples.

9.4.2.1 Some examples are: (1) total solids and total volatile solids are often done as one analysis. Total solids should be larger than or equal to the volatile component; (2) the ammonia nitrogen should always be equal to or less than Kjeldahl nitrogen, and (3) in specific treatment processes, the input sample results should always be equal to or greater than the result on the output sample.

9.4.2.2 Many comparisons similar to those listed in 9.4.2.1 can be made to ensure that data are logically consistent.

9.5 Where there is sufficient historical data or the expected analytical concentrations are supplied by the client, a reasonableness test of the analysis should be done during the review process. The analysis and the reviewer should determine if the results are close to the expected value. If the data are not within reasonable limits, the analytical method and calculations should be reviewed for deviations or anomalies. Contact with the customer should take place if no errors are found in the laboratory process.

9.6 A quality assurance narrative should be used to explain any discrepancies in the data or unusual conditions that resulted in data of questionable quality (that is, matrix interferences, elevated detection limits, and so forth).

9.7 The report should include the signature and title of the individual who verified the reported data before their release and verified that these results met the customer’s data quality specifications.

10. Reporting Low-level Data Concentrations

10.1 Some information is lost to the customer when the results are reported as “less than” or “below the criterion of detection” when there was an instrument response indication that there was something present. The customer should be allowed to make his own decision regarding the usefulness of

⁹ Friedman, L. C., and Erdmann, D. E., “Quality Assurance Practices for the Chemical and Biological Analyses of Water and Fluvial Sediments,” *Techniques of Water-Resources Investigations of the U.S. Geological Survey*, Book 5, Chapter A6, U.S. Government Printing Office, 1982.

¹⁰ Hem, J. D., “Study and Interpretation of the Chemical Characteristics of Natural Water,” *U.S. Geological Survey Water-Supply Paper 2254*, 1985.

TABLE 4 Effects of Censored and Uncensored Data, µg

Using less than (<)		Delete (–)		Uncensored Data
Column 1	2	3	4	5
LRL (3)	<as 0	½ LRL		
<3	0	1.5	2	2
<3	0	1.5	0	–2
<3	0	1.5	0	–1
4	4	4	4	4
3	3	3	3	3
<3	0	1.5	0	–3
<3	0	1.5	1	1
<3	0	1.5	0	–1
<3	0	1.5	0	0
<3	0	1.5	2	2

such data (see 8.1). The laboratory should have a standard policy for releasing data that are reported as “less than” or the criterion of detection.

10.2 In answering the question “Is a substance present?”, there are two possible correct conclusions that can be reached. One may conclude that the substance is present when it is present, and one may conclude that the substance is not present when it is not present. Conversely, there are two possible erroneous conclusions which may be reached. One may conclude that the substance is present when it is not (Type I error) and one may conclude that the substance is not present when it is (Type II error). However, if all data are reported, the customer can evaluate the data and come to his own conclusion based on their in-depth knowledge of the process stream or waste site. The analyst has the responsibility to place qualifiers on the data to ensure sufficient communication to occur to minimize misuse of data. The use of qualifiers does not negate the need for direct communication with the customer.

10.3 It is possible, when the analysis is at the detection limit, to have negative values when the blank is subtracted from the sample. If the constituent of interest is not present, one would expect negative results to occur as often as positive in the case of blank subtraction. Negative results are also possible when background data are subtracted from the sample data.

NOTE 6—Blank subtraction should not be done unless called for in the method or other extenuating circumstances occur which make subtraction necessary. See 8.8 for further discussion.

10.4 In Table 4, are listed data and five ways the data is reported. Depending upon how the data is presented, different conclusions can be drawn.

10.4.1 In Column 1 of Table 4, the laboratory reported the analysis using the less than a stated value (<3), the user may think the analyst did not know whether a compound was present or not. Such data are considered to be “censored” since some of the information has not been passed on the user.

10.4.2 In Column 2 of Table 4, the less than values have been reported as 0. The user now feels that they have an average value of 0.7, but eight out of the ten samples do not contain the compound(s) of interest.

10.4.3 In Column 3 in Table 4, the laboratory reports data below its laboratory reporting limit (LRL) as one half the the laboratory reporting limit. The mean of the results would be 1.9

µg. The data user would erroneously conclude that the compound was found in each of the samples.

10.4.4 The censored results found in the Column 4 in Table 4, were taken as reported without consideration of negative results, one would conclude that the mean concentration was 1.2 µg with a standard error of the mean of 0.467 and 95 % confidence limits for the mean of 0.14 and 2.26 µg. Since the confidence limits do not include zero, it would appear that the evidence supports the presence of the constituent.

10.4.5 Analysis of the uncensored results of Column 5 in Table 4 gives a mean concentration of 0.5 µg, a standard error

of the mean of 0.719 and 95 % confidence limits for the mean of -1.13 µg and 2.13 µg. The correct conclusion can be drawn that the evidence is insufficient to support the presence of the constituent.

10.4.6 The following data are taken from Practice D 4210 with some modification to illustrate each of the five points:

11. Keywords

11.1 analysis; blank; low-level reporting; reporting data; results; water

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