



Standard Guide for Planning, Carrying Out, and Reporting Traceable Chemical Analyses of Water Samples¹

This standard is issued under the fixed designation D 6568; 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 guide sets a protocol for generating and reporting chemical analyses that are traceable to SI Units or to Certified Reference Materials in laboratories that serve the water and environmental industry.

1.2 *This guide does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 1129 Terminology Relating to Water

D 6362 Standard Practice for Certificates of Reference Materials for Water Analysis

IEEE/ASTM SI 10–1997 Standard for use of the International System of Units (SI): the modern metric system

2.2 Other Documents:

ISO Guide 17025: General Requirements for the Competence of Calibration and Testing Laboratories²

ISO Guide 30: Terms and definitions used in connection with reference materials²

International Vocabulary of Basic and General Terms in Metrology [VIM]: ISO: 2nd ed., 1993²

3. Terminology

3.1 *Definitions*—For definitions of terms used in this Guide, refer to D 1129, Terminology Relating to Water.

3.1.1 *certified reference material*—reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which established its traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence. (ISO Guide 30:1992) (Note 1).

¹ This guide is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.02 on General Specifications, Technical Resources, and Statistical Methods.

Current edition approved July 10, 2000. Published October 2000.

² Available from American National Standards Institute, 11 West 42nd St., 13th Floor, New York, New York, 10036

NOTE 1—There is significant variation in the overall quality of commercially available Certified Reference Materials and caution should be used when choosing Certified Reference Materials. Use ASTM D 6362 to provide guidance as to what information needs to be included on Certificate of a Certified Reference Material.

3.1.2 *traceability*—property of the result of a measurement or the value of a standard whereby it can be related, with a stated uncertainty, to stated references, usually national or international standards, through an unbroken chain of comparisons. (ISO Guide 30:1992).

3.1.3 *uncertainty (of measurement)*—parameter, associated with the result of a measurement that characterizes the dispersion of values that could reasonably be attributed to the measured. (International Vocabulary of Basic and General Terms in Metrology [VIM] (ISO: 2nd ed., 1993).

3.1.4 *work plan*—a documented procedure intended for use by a laboratory to meet the measurement traceability requirements of a defined need.

3.1.5 *SI units*—this is the International System of Units (SI) which is the modernized Metric System as described in IEEE/ASTM SI 10-1997. A SI Quick Reference Guide is included at the back (in the “gray pages”) of volumes 11.01 and 11.02.

4. Significance and Use

4.1 This Guide establishes basic requirements which should be met by water and environmental laboratories that generate and report test chemical analyses which the laboratory client desires to be traceable to SI Units (Note 2) or Certified Reference Materials traceable to SI Units. Traceability of chemical analyses is important because it provides a uniform basis for the comparison of results from different measurement systems and because it relates those results to our current knowledge of physical laws. (Note 3)

NOTE 2—A Certified Reference Material traceable to SI Units is a Certified Reference Material whose value can be related with a stated uncertainty through an unbroken change of comparisons to stated references (usually national or international standards) in SI Units, such as a primary measurement made in SI Units or a national standard certified in SI Units.

NOTE 3—Not all chemical analysis results can be traceable to SI Units or to Certified Reference Material’s traceable to SI Units, such as Turbidity and or Total Suspended Solids.

4.2 Many waters-related laboratories comply with ISO Guide 17025 and participate in Proficiency Testing Programs. Laboratories that are connected to the same accreditation bodies and Proficiency Test providers can be expected to report statistically similar results on the same sample. However, some test methods and some Certified Reference Materials are not supported with data traceable to SI Units. Therefore, fully compliant laboratories that are not connected to the same providers may report statistically different chemical analysis results if they used the same nontraceable test method on the same sample. This problem could be minimized if they used test methods, measurement devices, and Certified Reference Materials that are traceable to SI Units, where available.

4.3 Although some standard test methods and Certified Reference Materials provide evidence of traceability to SI Units, many others do not. Therefore, not all laboratories can be expected to universally meet all requests for traceable analyses until the traceability of more test methods and Certified Reference Materials is recognized through appropriate documentation.

4.4 The primary significance of this Guide is that it establishes a consensus that, in order for a laboratory to generate traceable measurements, it must (1) have a clear understanding of the needs of the user of the traceable measurements, (2) comply with the internationally accepted quality-system requirements included in ISO Guide 17025, (3) use test methods, measurement devices, and certified reference materials which have been shown to be traceable to SI Units, and (4) be able to demonstrate that the measurement system was in statistical control at the time the measurements were made.

4.5 It is expected that this Guide will be used by Committee D-19 in setting policies for the technical content of its standards that are designated to be usable to generate traceable chemical analyses.

5. Summary

5.1 A client shall prepare a request for traceable chemical analyses and submit it to an ISO Guide 17025 compliant laboratory for review and acceptance. (Note 4)

NOTE 4—By reference, this Guide incorporates the laboratory quality system requirements of ISO Guide 17025. A laboratory that does not comply with ISO Guide 17025 (or an equivalent quality system guide such as the requirements document being produced by the National Environmental Laboratory Accreditation Council [NELAC]) is considered non-compliant with this Guide.

5.2 The laboratory shall prepare a work plan to meet the requirements and obtain concurrence with the client.

5.3 The laboratory shall make the chemical analyses and issue a report.

6. Procedure

6.1 The client shall prepare a request for traceable measurements and submit it to an ISO Guide 17025 compliant laboratory for review and acceptance, as follows.

6.1.1 The request shall identify the source (i.e. the specific sampling site, the process generating the sample, etc.), sampling history, configuration, and approximate composition of the test materials, the traceable measurements to be made with their required maximum uncertainties. It shall also identify

special requirements, such as, the need to certify that the material tested complies with a specific product specification or that traceability must be made to a specific standard or Certified Reference Material. The request shall also specify if any nontraceable chemical analyses are to be included as part of the project.

6.1.2 The client shall select an ISO Guide 17025 compliant laboratory and submit their request to the laboratory for review.

6.1.3 The laboratory shall review the request, work with the client to clarify their understanding of the need, and inform the client of its willingness and capability to proceed.

6.2 The laboratory shall prepare a work plan (Note 5) to meet the requirements and obtain concurrence with the client, as follows.

NOTE 5—A Work Plan can be in the form of a laboratory SOP that includes the requirements detailed in sections 6.2.1-6.2.6. This will allow a laboratory to have a “generic” Work Plan, which will reduce costs and increase efficiency.

6.2.1 The work plan shall identify protocols: for example, sample receipt, chain of custody, and sample preparation. It shall also identify all test methods to be used. All test methods used for traceable measurements shall have associated documentation showing their complete traceability to SI Units, where applicable. Traceability can be demonstrated either by direct measurement, use of pure materials which were assayed using methods traceable to SI Units, or use of Certified Reference Materials which were certified in a manner traceable to SI Units. If traceable standard methods are not available, the Laboratory may propose to validate new analytical methods for this work. The work plan shall also document how nontraceable measurements, if required, are to be carried out.

6.2.2 The work plan shall describe the anticipated measurement uncertainty for the project to ensure that the estimated uncertainties in the final report values do not exceed the client’s specified requirements. The statistical basis for the estimated uncertainty measurements described in the Work Plan should be identified, i.e. from in-house quality control data, from the precision and bias statement in the Standard Test Method, etc.

6.2.3 The work plan shall identify matrix-matched Certified Reference Materials that contain the relevant analyte at a concentration representative of the expected sample concentration (where available) or pure chemicals to be carried along with the traceable measurements. It shall also specify the maximum estimated uncertainties to be permitted in making these measurements. The laboratory staff shall make sure that the maximum estimated uncertainties, either determined under the Work Plan or published with a Certified Reference Material with similar matrix and analyte concentration (when available), are attainable and are small enough to permit the attainment of the expected values. (Note 6)

NOTE 6—The laboratory’s ability to meet these anticipated uncertainties will indicate that the measurement uncertainties in the measuring laboratory were reasonable while the traceable measurements were being carried out.

6.2.4 The Work Plan shall define the contents of the final report.

6.2.5 The work plan shall be reviewed and approved for presentation to the client by the laboratory's management. That review should include projected time and cost schedules, as needed, to meet local business needs.

6.2.6 The work plan shall be presented to the client for review. If the client decides that revisions are needed, those revisions shall be reviewed and approved by the laboratory's management before being presented to the client for final acceptance. The work plan shall be accepted by the client, after which the laboratory can begin to carry out the work.

6.3 The laboratory shall make the chemical analyses and issue a report, as follows.

6.3.1 The laboratory shall make the agreed-upon chemical analyses and document them in accordance with the work plan and ISO Guide 17025. If, in the course of making the measurements, it becomes necessary to deviate from the work plan in any way that will affect the client or the client's use of

the data, the laboratory shall inform the client. If necessary, the work plan shall be updated and approved as described in 6.2.

6.3.2 When the work is complete, Staff shall prepare and issue a written report that shall include all of the material and measurement traceability documentation described in the Work Plan. In the event that the work included any method validation (6.1.1) those results shall be included. The report shall also comply with the reporting requirements of ISO Guide 17025. (Note 7). It shall clearly differentiate between traceable and nontraceable measurements, if both were specified.

NOTE 7—It is recommended, but not required, that all copies of the original work plan be destroyed. Since the original work plan covered what was expected to occur and the final report describes what actually occurred, retaining the planning document may precipitate post-completion confusion about the integrity of the final report. If desired the Work Plan can be marked obsolete (or similar notation) and maintained.

7. Keywords

7.1 certified reference materials; measurement traceability

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).