Designation: D6800 – 18 (Reapproved 2026)

Standard Practice for Preparation of Water Samples Using Reductive Precipitation Preconcentration Technique for ICP-MS Analysis of Trace Metals[1]

This standard is issued under the fixed designation D6800; 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 Toxic elements may be present in ambient waters and may enter the food chain via uptake by plants and animals; the actual concentrations of toxic metals are usually sub-ng/mL. The U.S. EPA has published its Water Quality Standards in the U.S. Federal Register 40 CFR 131.36, Minimum requirements for water quality standards submission, Ch. I (7-1-00 Edition), see Annex, Table A1.1. The U.S. EPA has also developed Method 1640 to meet these requirements, see Annex, Table A1.2.

1.2 Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) is a technique with sufficient sensitivity to routinely measure toxic elements in ambient waters, both fresh and saline (Test Method D5673). However saline and hard water matrices pose analytical challenges for direct multielement analysis by ICP-MS at the required sub-ng/mL levels.

1.3 This practice describes a method used to prepare water samples for subsequent multielement analysis using ICP-MS. The practice is applicable to seawater and fresh water matrices, which may be filtered or digested. Samples prepared by this method have been analyzed by ICP-MS for the elements listed in Annex, Table A1.3).

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D5673 Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry
D5810 Guide for Spiking into Aqueous Samples
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

2.2 *Other Documents:*

U.S. Federal Register 40 CFR 131.36, Minimum Requirements for Water Quality Standards Submission, Ch. I (7-1-00 Edition)[3]
U.S. EPA Method 1640, Determination of Trace Elements in Water by Preconcentration and Inductively Coupled Plasma-Mass Spectrometry (1997)[4]
U.S. EPA Method 1669, Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels[4]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *continuing calibration blank, n*—a solution containing no analytes (of interest) which is used to verify blank response and freedom from carryover.

[1] This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

Current edition approved May 1, 2026. Published June 2026. Originally approved in 2002. Last previous edition approved in 2018 as D6800 – 18. DOI: 10.1520/D6800-18R26.

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

[3] Available from DLA Document Services, Building 4/D, 700 Robbins Ave., Philadelphia, PA 19111-5094, http://quicksearch.dla.mil.

[4] Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

3.2.2 *continuing calibration verification, n*—a solution (or set of solutions) of known concentration used to verify freedom from excessive instrumental drift; the concentration is to cover the range of calibration curve.

3.2.3 *intermediate stock-standard solution, n*—a diluted solution prepared from one or more of the stock-standard solutions.

3.2.4 *laboratory control sample (LCS), n*—an aliquot of solution with known concentrations of method analytes.

3.2.4.1 *Discussion*—The LCS should be obtained from a reputable source or prepared at the laboratory from a separate source from the calibration standards. The LCS is analyzed using the same sample preparation, analytical method and QA/QC procedure used for test samples. Its purpose is to determine whether method performance is within accepted control limits.

3.2.5 *laboratory duplicate (LD), n*—a second sample aliquot that is analyzed using the same sample preparation, analytical method and QA/QC procedure used for test samples.

3.2.5.1 *Discussion*—The purpose of the LD is to determine whether method performance is within accepted control limits.

3.2.6 *matrix spike (MS), n*—a second sample aliquot to which known concentrations of target analyte(s) are added in the laboratory and which are analyzed using the same sample preparation and analytical method used for test samples.

3.2.6.1 *Discussion*—The purpose of the MS is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of the matrix must be determined in a separate aliquot and the measured values in the MS corrected for the concentrations found. Recommended spike levels are listed in Annex, Table A1.3.

3.2.7 *method blank (MB), n*—suitable reagent-water aliquots that are analyzed using the same sample preparation, analytical method, and QA/QC procedure used for test samples.

3.2.7.1 *Discussion*—The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or apparatus.

3.2.8 *method detection limit (MDL), n*—a limit determined as described in the U.S. Federal Register (see 40 CFR Part 136, Appendix B).

3.2.9 *reagent water, n*—standard laboratory water purified to meet Specification D1193 Type I or better.

3.2.10 *reporting detection limit (RDL), n*—the lowest concentration at which an analyte can be reliably quantified.

3.2.10.1 *Discussion*—The RDL represents the minimum concentration at which method performance becomes quantitative and is not subject to the degree of variation observed at concentrations between the MDL and the RDL

3.2.11 *spiked blank (SB), n*—a reagent-water aliquot to which known concentrations of analyte(s) are added in the laboratory, using the same solution as used to prepare the matrix spike.

3.2.11.1 *Discussion*—The spike blank is analyzed using the same sample preparation, analytical method and QA/QC procedure used for test samples. The purpose of the spike blank is to determine whether method performance is within acceptable limits. The spike blank is also useful for troubleshooting matrix-spike results that are outside the acceptance limits, by allowing the analyst to differentiate between: (*1*) spike-solution and spiking-technique problems, and (*2*) matrix interferences. Recommended spike levels are listed in Annex, Table A1.3.

3.2.12 *stock-standard solution, n*—a concentrated solution (containing one or more analytes), obtained as a certified solution from a reputable source.

3.2.13 *surrogate spikes, n*—lanthanum and terbium are added at a concentration of 5 ng/mL (each) in the initial 100 mL sample.

3.2.13.1 *Discussion*—The surrogate spikes are then preconcentrated to approximately 50 ng/mL in the final 10 mL sample without correcting for the final preconcentration. The surrogate spikes are used to determine potential method problems such as improper pH adjustment or faulty filters used when collecting the precipitate.

3.2.14 *total recoverable, n*—the concentration of analyte determined on a whole, unfiltered water or solid sample following vigorous digestion as described in U.S. EPA Method 1640.

4. Summary of Practice

4.1 In this practice, trace elements are separated from seawater matrix elements (in particular Na, Ca, and Mg) and preconcentrated by a factor of 10 by reductive precipitation using sodium borohydride as a reducing agent.

4.2 Iron (Fe) and palladium (Pd) are added to the samples to aid co-precipitation of metal borides and to enhance the precipitation of metals in their elemental form.

4.3 For total metals, the whole sample is acidified at the time of collection with ultrapure nitric acid at an equivalent concentration of 0.20 % to a pH < 2.

4.4 For dissolved metals, the sample is filtered through a 0.4 µm filter at the time of collection then acidified with ultrapure nitric acid at an equivalent concentration of 0.20 % to a pH < 2.

NOTE 1—It is important to minimize the amount of nitric acid used to preserve the samples. A pH adjustment to a pH between 8 and 10 using ammonium hydroxide is performed during the co-precipitation reaction and it is important to minimize the amount of ammonium hydroxide required for this adjustment to reduce potential contamination of the samples.

4.5 The precipitate is collected by filtration through a 0.4 µm filter and the salt matrix is eliminated with the filtrate. The filter and precipitate are digested with nitric acid and hydrogen peroxide for analysis by ICP-MS.

5. Significance and Use

5.1 Ambient marine waters generally contain very low concentrations of toxic metals that require sensitive analytical methods, such as ICP-MS, to detect and measure the metal's concentrations.

5.2 Due to the high dissolved salt concentrations present in seawater, sample pretreatment is required to remove signal