



Designation: ~~D4453~~—~~11~~ D4453 – 16

Standard Practice for Handling of High Purity Water Samples¹

This standard is issued under the fixed designation D4453; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This practice² covers concepts for handling high purity water samples needed for the measurement of ever-decreasing levels of specified impurities that are encountered in the operation of modern high-pressure boilers and turbines. The handling of blanks associated with the analysis of high purity water samples is also covered by this practice. The techniques presented can help the investigator increase the accuracy of analyses performed.

1.2 This practice is applicable to water and steam samples from “zero solids treated” once-through or drum-type boilers, reactor coolant water, electronic grade water, or any other process water where analyte concentrations are in the low parts per billion (micrograms per litre) range.

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

1.4 *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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in 6.2.3.5, 6.1, and 6.3.7.

2. Referenced Documents

2.1 *ASTM Standards:*³

[D1066 Practice for Sampling Steam](#)

[D1129 Terminology Relating to Water](#)

[D1193 Specification for Reagent Water](#)

3. Terminology

3.1 ~~Definitions—Definitions:~~ For definitions of terms used in these test methods, refer to Terminology [D1129](#).

3.1.1 For definitions of terms used in this standard, refer to Terminology [D1129](#).

4. Significance and Use

4.1 The determination of trace impurities (on the order of parts per billion) in high purity water places extreme requirements on all aspects of the analytical system. This is particularly true when ubiquitous species such as sodium and chloride are of interest because they can potentially be introduced as contaminants at almost every step of an analytical procedure. Contamination can occur during sample collection, during sample storage by leaching of improperly cleaned containers, during sample transfer, and by handling with pipets, syringes, etc., and during the actual analysis by contaminated reagents and sample cells and loop systems. It is also possible that trace contaminants can be lost from samples by volatilization or precipitation, by diffusion into the matrix of the container material, and by “plating out” on the walls of sampling lines by flow phenomena.

4.2 Strict adherence to a given procedure is necessary to achieve good results at trace levels of analysis because very small differences in procedure execution will affect precision and the addition or loss of nanogram amounts of analyte may affect the accuracy of a determination.

¹ This practice is under the jurisdiction of ASTM Committee [D19](#) on Water and is the direct responsibility of Subcommittee [D19.03](#) on Sampling Water and Water-Formed Deposits, Analysis of Water for Power Generation and Process Use, On-Line Water Analysis, and Surveillance of Water.

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² This practice suggests the use of specific techniques. As new techniques are developed or required by lower limits, revision of this practice will likely be needed.

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.

5. Reagents and Materials

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. It is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Reference to water that is used for reagent preparation, rinsing or dilution shall be understood to mean water that conforms to the quantitative specifications of Type II reagent water of Specification **D1193**.

5.3 *Hydrochloric Acid (1 + 1)*—Dilute concentrated hydrochloric acid with an equal volume of water.

5.4 *Nitric Acid (1 + 1)*—Dilute concentrated nitric acid with an equal volume of water.

5.5 *Nitric Acid*, ultra-pure.

5.6 *Methanol*.

5.7 *n-hexane*.

5.8 *Nitrogen*, organic-free.

6. Procedure

6.1 *Environmental Conditions*—Any processes that might contaminate the analytes should be excluded from the sampling and handling area, e.g., for example, smoke, volatile organic solvents, etc. (**Warning**—When performing trace organic analyses, do not allow the use of volatile organic solvents or smoking at the same time and within the area. ~~Warning~~—when performing trace organic analyses, do not allow the use of volatile organic solvents or smoking at the same time and within the area.)

6.2 *Sample Containers and Their Treatment:*

6.2.1 *Analysis of Trace Metals*⁵:

6.2.1.1 Bottles made from the following materials should be adequate: TFE-fluorocarbon FEP, HDPE, LDPE, Polypropylene, and polycarbonate. Caps should be made of the same material or, if not available, the caps should be lined with one of the suggested materials.

6.2.1.2 Fill the bottle with HCl (1 + 1) and allow to stand for 48 h at room temperature (80°C for TFE-fluorocarbon), then empty and rinse with water.

6.2.1.3 Fill the bottle with HNO₃ (1 + 1) and allow to stand for 48 h at room temperature (80°C for TFE-fluorocarbon), then empty and rinse with water.

6.2.1.4 Fill the bottle about one third full with water, cap, shake, and empty. Wear disposable polyethylene gloves when handling the bottles from this point onward. If a contaminated surface is touched, remove and replace the glove.

6.2.1.5 Repeat 6.2.1.4.

6.2.1.6 Completely fill the bottle with water, cap, and allow to soak for 48 h. After filling, place the bottle in a polyethylene bag and seal the bag during the storage period. The water should be changed at weekly intervals during long storage periods.

6.2.2 *Analysis of Other Trace Ions*⁶:

6.2.2.1 Containers made from the following materials should be adequate: HDPE, LDPE, polysulfone, polycarbonate, polystyrene, polypropylene, and polymethyl pentene. The temperature of the samples should not be above 50°C when taken or during the storage period to prevent leaching from the containers. (See 6.3.3.)

6.2.2.2 Fill the bottle one third full with water, cap, shake, and empty. Wear disposable polyethylene gloves when handling the bottles from this point onward. If a contaminated surface is touched, remove and replace the glove.

6.2.2.3 Repeat 6.2.2.2.

6.2.2.4 Completely fill the bottle with water, cap, and allow to soak for a minimum of 1 day and a maximum of 5 days. After filling, place the bottle in a polyethylene bag and seal the bag during the storage period. The water should be changed at 5 day intervals during long storage periods.

6.2.3 *Analysis of Trace Organics (Acid and Base Neutrals):*

6.2.3.1 The sample bottle must be borosilicate glass with a TFE-fluorocarbon-lined or aluminum-lined cap.

6.2.3.2 Wash in hot detergent water and rinse with tap water three times.

6.2.3.3 Rinse three times with water.

6.2.3.4 Bottles must be either heated to a minimum of 400°C in a muffle furnace (or dry heat sterilizer) for at least 30 min or solvent rinsed as in 6.2.3.5.

6.2.3.5 Rinse with solvents in the following order: methanol, n-hexane. Gloves worn during the solvent rinse may contain plasticizers (phthalates) that may cause contamination. Polyurethane, polyethylene, or nitrile rubber gloves are not likely to have

⁴ "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analytical Standards for Laboratory Chemicals," BDH Ltd., Poole, Dorset, U.K., and the "United States Pharmacopeia."

⁵ Except alkali and alkali earth metals.

⁶ Ions such as Na⁺, K⁺, Cl⁻, Ca²⁺, Mg²⁺, F⁻, Br⁻, NH₄⁺, NO₃⁻, SO₄²⁻, and PO₄³⁻.