



Designation: D 4453 – 02

Standard Practice for Handling of Ultra-Pure Water Samples¹

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1. Scope

1.1 This practice² covers concepts for handling ultra-pure 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 ultra-pure 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 *This standard does not purport to address all of the safety problems, 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 5.2.3.5, 5.1, and 5.3.7.

2. Referenced Documents

2.1 ASTM Standards:

D 1066 Practice for Sampling Steam³

D 1193 Specification for Reagent Water³

3. Significance and Use

3.1 The determination of trace impurities (on the order of parts per billion) in ultra-pure 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.

3.2 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.

4. Reagents and Materials

4.1 *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.

4.2 *Purity of Water*—Reference to high-purity water shall be understood to mean water conforming to Specification D 1193, Type I reagent water, post treated with an organic removal cartridge or demineralized water which has additionally been polished using a cartridge water purification system with an organic removal cartridge and 0.2- μ m final filter.

4.3 *Hydrochloric Acid (1 + 1)*—Dilute concentrated hydrochloric acid with an equal quantity of high purity water.

4.4 *Nitric Acid (1 + 1)*—Dilute concentrated nitric acid with an equal quantity of high purity water.

4.5 *Nitric Acid*, ultra-pure.

4.6 *Methanol*.

4.7 *n-hexane*.

¹ This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.03 on Sampling of Water and Water-Formed Deposits, Surveillance of Water, and Flow Measurement 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 *Annual Book of ASTM Standards*, Vol 11.01.

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.