



# Standard Specification for Reagent Water<sup>1</sup>

This standard is issued under the fixed designation D 1193; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

## 1. Scope

1.1 This specification describes the required characteristics of waters deemed suitable for use with the Standards under the jurisdiction of ASTM.

1.2 The alphanumeric characters ascribed to water types and grades are specified in the ASTM Format and Style Manual. These have been assigned in order of historical precedence and should not be taken as an indication of a progression in water purity.

1.3 Four types of waters have been specified, with three additional grades that can be applied to the four types. The grade specifications specifically address contaminants of microbiological origin.

1.4 All applicable ASTM Standards are expected to reference one or more of these reagent water types where reagent water is needed as a component of an analytical measurement process. Where a different water type or grade is needed for an ASTM Standard, it may be added to this Specification through the ASTM Standard revision process.

1.5 Although these water types and associated grades have been defined specifically for use with ASTM Standards, they may be appropriate for other applications. It is the responsibility of the users of this standard to ensure that the selected water types or grades are suitable for their intended use. Historically, reagent water Types I, II, III, and IV have been linked to specific processes for their production. Starting with this revision, these types of waters may be produced with alternate technologies as long as the appropriate constituent specifications are met *and that water so produced has been shown to be appropriate for the application where the use of such water is specified*. Therefore, the selection of an alternate technology in place of the technology specified in **Table 1** should be made taking into account the potential impact of other contaminants such as microorganism and pyrogens. Such contaminants were not necessarily considered by the performance characteristics of the technology previously specified.

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee D19 on Water and is the responsibility of Subcommittee D19.02 on General Specifications, Technical Resources, and Statistical Methods.

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1.6 Guidance for applications, the preparation, use and monitoring, storage, handling, distribution, testing of these specified waters and validation of the water purification system is provided in **Appendix X1** of this document.

1.7 *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.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

**D 1125** Test Methods for Electrical Conductivity and Resistivity of Water

**D 1129** Terminology Relating to Water

**D 1293** Test Methods for pH of Water

**D 4453** Practice for Handling of Ultra-Pure Water Samples

**D 4517** Test Method for Low-Level Total Silica in High-Purity Water by Flameless Atomic Absorption Spectroscopy<sup>3</sup>

**D 5128** Test Method for On-Line pH Measurement of Water of Low Conductivity

**D 5173** Test Method for On-Line Monitoring of Carbon Compounds in Water by Chemical Oxidation, by UV Light Oxidation, by Both, or by High Temperature Combustion Followed by Gas Phase NDIR or by Electrolytic Conductivity

**D 5245** Practice for Cleaning Laboratory Glassware, Plasticware, and Equipment Used in Microbiological Analyses

**D 5391** Test Method for Electrical Conductivity and Resistivity of a Flowing High Purity Water Sample

**D 5542** Test Method for Trace Anions in High Purity Water by Ion Chromatography

**D 5997** Test Method for On-Line Monitoring of Total Carbon, Inorganic Carbon in Water by Ultraviolet, Persulfate

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Determination of Trace Silica in Industrial Process Waters by Flameless Atomic Absorption Spectrometry, Judith Rawa and Earl Henn, *Analytical Chemistry*, Vol 51, No 3, March 1979.

**TABLE 1 Processes for Reagent Water Production**

Type	Grade	Production Process <sup>A,B,C,D</sup>	µS/cm <sup>E</sup> (max)	MΩ·cm <sup>F</sup> (min)	pH <sup>G</sup>	TOC µg/L <sup>H</sup> (max)	Sodium µg/L <sup>I</sup> (max)	Chloride µg/L <sup>J</sup> (max)	Total Silica µg/L (max)	HBC <sup>K</sup> cfu/mL (max)	Endotoxin, EU/mL <sup>L</sup> (max)
I		Purify to 20 µS/cm by dist. or equiv., followed by mixed bed DI, 0.2 µm filtration <sup>A</sup>	0.0555	18		50	1	1	3		
I	A	Purify to 20 µS/cm by dist. or equiv., followed by mixed bed DI, 0.2 µm filtration <sup>A</sup>	0.0555	18		50	1	1	3	10/1000	0.03
I	B	Purify to 20 µS/cm by dist. or equiv., followed by mixed bed DI, 0.2 µm filtration <sup>A</sup>	0.0555	18		50	1	1	3	10/100	0.25
I	C	Purify to 20 µS/cm by dist. or equiv., followed by mixed bed DI, 0.2 µm filtration <sup>A</sup>	0.0555	18		50	1	1	3	100/10	
II		Distillation <sup>B</sup>	1.0	1.0		50	5	5	3		
II	A	Distillation <sup>B</sup>	1.0	1.0		50	5	5	3	10/1000	0.03
II	B	Distillation <sup>B</sup>	1.0	1.0		50	5	5	3	10/100	0.25
II	C	Distillation <sup>B</sup>	1.0	1.0		50	5	5	3	100/10	
III		Distillation, DI, EDI, and/or RO, followed by 0.45 µm filtration. <sup>C</sup>	0.25	4.0		200	10	10	500		
III	A	Distillation, DI, EDI, and/or RO, followed by 0.45 µm filtration. <sup>C</sup>	0.25	4.0		200	10	10	500	10/1000	0.03
III	B	Distillation, DI, EDI, and/or RO, followed by 0.45 µm filtration. <sup>C</sup>	0.25	4.0		200	10	10	500	10/100	0.25
III	C	Distillation, DI, EDI, and/or RO, followed by 0.45 µm filtration. <sup>C</sup>	0.25	4.0		200	10	10	500	1000/100	
IV		Distillation, DI, EDI, and/or RO. <sup>D</sup>	5.0	0.2	5.0 to 8.0		50	50			
IV	A	Distillation, DI, EDI, and/or RO. <sup>D</sup>	5.0	0.2	5.0 to 8.0		50	50		10/1000	0.03
IV	B	Distillation, DI, EDI, and/or RO. <sup>D</sup>	5.0	0.2	5.0 to 8.0		50	50		10/100	0.25
IV	C	Distillation, DI, EDI, and/or RO. <sup>D</sup>	5.0	0.2	5.0 to 8.0		50	50		100/10	

<sup>A</sup> Type I grade of reagent water shall be prepared by distillation or other equal process, followed by polishing with a mixed bed of ion-exchange materials and a 0.2-µm membrane filter. Feed water to the final polishing step must have a maximum conductivity of 20 µS/cm at 298K (25°C). Type I reagent water may be produced with alternate technologies as long as the appropriate constituent specifications are met *and that water so produced has been shown to be appropriate for the application where the use of such water is specified.*

<sup>B</sup> Type II grade of reagent water shall be prepared by distillation using a still designed to produce a distillate having a conductivity of less than 1.0 µS/cm at 298 K (25°C). Ion exchange, distillation, or reverse osmosis and organic adsorption may be required prior to distillation, if the purity cannot be attained by single distillation. Type II reagent water may be produced with alternate technologies as long as the appropriate constituent specifications are met *and that water so produced has been shown to be appropriate for the application where the use of such water is specified.*

<sup>C</sup> Type III grade of reagent water shall be prepared by distillation, ion exchange, continuous electrodeionization, reverse osmosis, or a combination thereof, followed by polishing with a 0.45-µm membrane filter. Type III reagent water may be produced with alternate technologies as long as the appropriate constituent specifications are met *and that water so produced has been shown to be appropriate for the application where the use of such water is specified.*

<sup>D</sup> Type IV grade of reagent water may be prepared by distillation, ion exchange, continuous electrodeionization, reverse osmosis, electro dialysis, or a combination thereof. Type IV reagent water may be produced with alternate technologies as long as the appropriate constituent specifications are met *and that water so produced has been shown to be appropriate for the application where the use of such water is specified.*

<sup>E</sup> Electrical conductivity at 25°C.

<sup>F</sup> Electrical resistivity at 25°C.

<sup>G</sup> pH at 25°C, not applicable to higher resistivity waters.

<sup>H</sup> Total organic carbon.

<sup>I</sup> Sodium.

<sup>J</sup> Chloride ion.

<sup>K</sup> Heterotrophic bacteria count.

<sup>L</sup> Endotoxin in endotoxin units per mL.

### Oxidation and Membrane Conductivity Detection

**D 6071** Test Method for Low Level Sodium in High Purity Water by Graphite Furnace Atom Absorption Spectroscopy

**D 6161** Terminology Used for Crossflow Microfiltration, Ultrafiltration, Nanofiltration and Reverse Osmosis Membrane Processes

**D 6529** Test Method for Operating Performance of Continuous Electrodeionization Systems on Feeds from 50–1000 µS/cm

**F 1094** Test Methods for Microbiological Monitoring of Water Used for Processing Electron and Microelectronic Devices by Direct Pressure Tap Sampling Valve and by the Presterilized Plastic Bag Method

### 3. Terminology

3.1 *Definitions*— For definitions used in this specification refer to Terminology **D 1129**.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *reagent water*—water that is used specifically as a component of an analytical measurement process and meets or exceeds the specifications for these waters.

3.2.2 *electrodeionization*—a process that removes ionized and ionizable species from liquids using electrically active media and using an electrical potential to influence ion transport, where the ionic transport properties of the active media are a primary sizing parameter. Electrodeionization

devices typically comprise semi-permeable ion-exchange membranes and permanently charged ion-exchange media (see Test Method **D 6529**).

3.2.3 *reverse osmosis (RO)*—the separation process where one component of a solution is removed from another component by flowing the feed stream under pressure across a semipermeable membrane. RO removes ions based on electrochemical forces, colloids, and organics down to 150 molecular weight. May also be called hyperfiltration (see Terminology **D 6161**)

#### 4. Composition and Characteristics

4.1 The types and grades of water specified in this Standard shall conform to the requirements in **Table 1**.

#### 5. Test Methods

5.1 *Electrical Conductivity and Resistivity*—Refer to Test Methods **D 1125** and **D 5391**.

5.2 *pH*—Refer to Test Methods **D 1293** and **D 5128**.

5.3 *Silica*—Refer to Test Method **D 4517**.

5.4 *Sodium*—Refer to Test Methods **D 6071**.

5.5 *Chlorides*—Refer to Test Method **D 5542**.

5.6 *TOC*—Refer to Test Methods **D 5173** and **D 5997**.

5.7 *Endotoxins*—Refer to LAL Test Method.<sup>4</sup>

5.8 *Microbiological Contamination*—Refer to Test Methods **F 1094**.

#### 6. Keywords

6.1 laboratory analysis; reagent; water

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<sup>4</sup> Published in the *U.S. Pharmacopeia* by The U.S. Pharmacopeial Convention, Inc.

## APPENDIX

### (Nonmandatory Information)

#### X1. POTENTIAL REAGENT WATER ISSUES

##### INTRODUCTION

This Appendix is provided as a guide to various issues in the production, application, storage, and monitoring of Reagent Water. These issues are very complex and extensive. This guidance is not intended to be comprehensive or complete. Producers and users of Reagent Water are encouraged to seek out additional sources of guidance in this area.

##### X1.1 Preparation

X1.1.1 Historically, reagent water Types I, II, III, and IV have been linked to specific process for their production. Starting with this revision, these types of waters may be produced with alternate technologies as long as the appropriate constituent specifications are met *and that water so produced has been shown to be appropriate for the application where the use of such water is specified*.

X1.1.2 The preparation methods of the various grades of reagent water influences the limits of impurities. Therefore, the selection of an alternate technology in place of the technology specified in the **Table 1** should be made taking into account the potential impact of other contaminants such as micro-organism and pyrogens, even if a grade is not specified. Such contaminants were not necessarily considered by the performance characteristics of the technology previously specified.

##### X1.2 Use and Application

###### X1.2.1 *Type I and Type III Water:*

X1.2.1.1 Contact with the ion-exchange materials may cause an addition of organic contaminants to the water. This will depend on the resin type/quality, quality of the regenerations (if regenerated), environmental conditions in which the water purification system is used and actual system use (for

example, duration of non-use periods). Practices may be put in place to decrease the risk of organic contamination:

(1) Periodic rinsing of the purification media to limit bacteriological (organic) contamination is recommended.

(2) After each period of non-usage, drawing off a quantity of water is necessary before use. Refer to the supplier specifications for the recommended volume.

(3) Synthetic activated carbon and/or UV (dual wavelengths 185 nm and 254 nm) may be used in the polishing stages to decrease the level of organic contaminants (to reach Type I water specifications), and/or to reach lower organic levels.

X1.2.1.2 The quality of the water produced depends upon the type, age, and method of regeneration of the ion exchange materials (if regenerated). Likewise, the flow rate through the ion exchange resin bed will change the conductivity of the product water. The manufacturer's instructions for resins or the resin cartridge bed should be followed.

X1.2.1.3 The use of the membrane filter in the preparation of Type I and Type III water may add a small amount of organic components to the water initially produced. The amount of organic components released differs depending on the type and brand of the membrane filter used. Then the membrane should be rinsed according to the manufacturer's

instructions. The use of a qualified membrane filter on the organic release is recommended.

X1.2.1.4 Producing Type I water specifications is achieved utilizing a combination of purification technologies. The choice of technologies can vary depending on the feed water quality, system usage and cost considerations. Particular attention should be taken regarding the location and sequence of particular purification technologies in the process, as these can have an impact on the final water quality.

#### X1.2.2 *Type II Water:*

X1.2.2.1 The description of Type II reagent water was intended to characterize product water from distillation processes. Therefore, the selection of an alternate technology in place of the one specified should be made by taking into account the potential impact of other contaminants (such as micro-organism and pyrogen) than those specified in **Table 1** for the Type II water.

X1.2.2.2 Type II grade of reagent water is typically sterile and pyrogen-free as produced and generally may be used whenever freedom from biological contaminants is desirable. However, the method of storage and handling of the water may itself result in contamination.

X1.2.2.3 Type II water is typically pyrogen-free as produced, but should be tested in conformance with the requirements of the referenced edition of *United States Pharmacopeia*, if proof is needed.

#### X1.2.3 *All Types of Water:*

X1.2.3.1 Biological contaminants may be important in the test procedure using any of the reagent waters specified. A classification of bacterial levels is included and should be specified if it is of significance to the test being performed.

X1.2.3.2 It should also be noted that the method used to prepare the different types of reagent water may or may not remove non-ionized dissolved gases. If non-ionized dissolved gases are of concern for the application considered, the selection of a method to produce water appropriate for the purpose and compliant with the **Table 1** specifications for the type and grade of water should be considered.

X1.2.3.3 To obtain sterile water, any of the types of reagent water listed in this section may be produced, bottled, and heated to 121°C for 20 min. This procedure is most easily carried out by autoclaving at 103 kPa (15 psi) for 20 min. Alternatively, sterilizing filtration of any types of the reagent water listed with a validated filter may also produce sterile water when performed in aseptic conditions. The user should choose the appropriate sterilization technique for the intended use.

### X1.3 Monitoring

X1.3.1 The limits of **Table 1** apply to the water sampled at the point of use or, when for practical reasons and/or to avoid contamination (for example connection of an equipment after a 0.2 µm filter), as close as possible to the point of use and with a regular verification of a low impact of the purification steps and/or equipment placed downstream of the monitoring sampling point.

X1.3.2 Because atmospheric gases and impurities rapidly recontaminate exposed water, an on-line sensor should be employed for determining the electrical conductivity of reagent

water Types I, II, and III. As atmospheric organic compounds and those from sampling vials rapidly contaminate exposed purified water, an on-line TOC monitor should be preferred for determining the TOC level of Type I and Type II water.

X1.3.3 Quality and system performance parameters should be regularly reported and registered. Follow-up of trends in the quality and performance parameters should be performed regularly to check any variations in performance of the water purification installation and to be able to anticipate any failures.

X1.3.4 The monitoring of different parameters should be performed at a frequency defined by the user to ensure with a high degree of confidence that the water quality used is always compliant with the specifications and the purpose.

### X1.4 Storage and Distribution

X1.4.1 Generally speaking, storage of the purified water will cause a loss of the specified characteristics. The impact will be different depending on the water type and grade produced (resistivity characteristics for example, will be rapidly impacted as soon as Type I water is stored). Water types, other than Type I, can be stored if particular attention is taken regarding the materials, design of the storage system and time of storage. The material of the storage reservoir in contact with water should be selected to minimize the release of extractables.

X1.4.1.1 The design of the storage reservoir should be fully drainable, either opaque or placed in an environment which limit bacterial growth by the effects of light.

X1.4.1.2 The storage container should be adequately protected from air contaminants (particles and CO<sub>2</sub>, especially when water is drawn) and from bacteriological contamination. This should be achieved by air filtration, inert gas blanketing, UV irradiation, chemical sanitization, heating above 80 degrees C, or a combination thereof. It should be recognized that the mere fact that the water is stored will likely reduce its purity despite attempts to prevent contamination. Storage should be sized to ensure a good turnover of water.

X1.4.1.3 Manual or automatic draw-off and periodic sanitization should be performed in particular after long periods of non-use. The periodicity of such draw-offs and sanitization should be defined by the user depending on the water purification system usage and water use. This periodicity can be defined during a qualification phase. After each sanitization, verification of the absence of the sanitization agent should be performed.

X1.4.2 If a distribution system is used to transfer the water to a laboratory, it should be of special design to minimize contamination. Gravity feed is the preferred method (if possible), since pumps are a potential source of contamination.

X1.4.2.1 If circulating systems are employed, the pumps should be designed to limit any contamination.

X1.4.2.2 The piping materials, fittings, faucets, and joints should be designed to limit any contamination.

X1.4.2.3 Outlets should be protected by UV or by micro-filtration (absolute 0.22 µm filter) or other means to prevent “back contamination” by airborne biological impurities.



X1.4.2.4 A loop distribution design is preferred to an antenna distribution, which can constitute a dead legs during periods of non-use.

X1.4.2.5 Positive pressure should be maintained in the distribution systems to avoid any retro-contamination.

X1.4.2.6 Microbiological proliferation should be minimized by suitable choice of periods of recirculation, flow rate, and/or temperature.

## **X1.5 Handling**

X1.5.1 Extreme care should be taken in handling reagent water during analyses. Depending on the water type required and the applications performed, container material and cleaning procedures must be chosen appropriately. Practices **D 5245** and **D 4453** should be consulted.

X1.5.2 Laboratory-ware should be carefully selected according to the application. Low release plastic-ware, such as PFA or TFE fluorocarbon (except for analysis of fluoride) or HDPE laboratory-ware should be used for ion-sensitive applications and high purity glass containers may be preferable for organic-sensitive applications.

## **X1.6 Maintenance and Calibration**

X1.6.1 Periodic calibration (if pertinent) of the different measuring instruments should be performed to ensure the validity of the values obtained.

X1.6.2 Periodic preventive maintenance should be performed to ensure the long-term performance and reliability of the water purification system. Follow-up of trends in the quality and performance parameters should be performed regularly to check any variations in performance of the installation and to be able to anticipate any failures.

X1.6.3 The frequency of system calibration and maintenance of the system should be defined by the user depending on the importance of the water in applications, but should not be performed less than once a year.

## **X1.7 Validation**

X1.7.1 Because quality assurance is the key to ensure safety, efficiency and reliability, validation is becoming increasingly important. The validation process can be divided into 4 major qualification steps:

X1.7.1.1 *Design Qualification (DQ)*—The Design Qualification is carried out before the selection of water purification system is made and consists of defining the water types

required depending on the applications, and defining the technology(ies) to be used, including the monitors to verify water quality. The design of the installation should also be defined according to requirements. All steps should be documented.

X1.7.1.2 *Installation Qualification (IQ)*—The Installation Qualification should take place after the installation of the system and consists of verifying and documenting that the installation was performed according to the predetermined specifications. This requires that the calibration of the various measuring instruments be verified. The actual installation should be compared with an installation drawing to ensure that no future installation modification be performed without suitable control management. Verification of the availability of all documentation required to use and maintain the system should also be done. Documented verification of the water purification system may be performed to ensure that the installation was performed according to specifications.

X1.7.1.3 *Operational Qualification (OQ)*—The Operational Qualification is performed after installation of the system and consists of ensuring that the system is operating according to the predetermined specifications. Tests should be conducted to verify that the hydraulic, monitoring and electronic functions (including system alerts) of the systems are working according to the specifications.

X1.7.1.4 *Performance Qualification (PQ)*—The Performance Qualification should be carried out after that the installation and operational qualification have been performed to document that the system is performing according to the predetermined specifications. During this qualification step verification of the appropriateness of the specifications, defined according to the applications, and verification of the water quality produced should be conducted.

X1.7.2 Re-qualification should be conducted on a regular time-basis and also each time components are replaced which can affect the quality or the quantity of water.

X1.7.2.1 The frequency of re-qualification depends on the importance of purified water in applications but cannot exceed one year. This ensures complete annual verification of the system alerts and calibration of the measuring instrument.

X1.7.2.2 A preventive maintenance (see Maintenance and Calibration section) should be conducted regularly and all actions should be documented in a dedicated system logbook.

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