



# Standard Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry<sup>1</sup>

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## 1. Scope

1.1 This guide provides recommendations for water quality related to current electronics and semiconductor industry requirements.

1.2 Water is used for washing and rinsing of semiconductor components during manufacture. It is also used for cleaning and etching operations, making steam for oxidation of silicon surfaces, photomask preparation and luminescent material deposition. Other applications are in the development and fabrication of solid state, thin film, communication lasers, light emitting diodes, photo-detectors, printed circuits, memory, vacuum tube, or electrolytic devices.

1.3 This guide also provides recommendations for ultra pure water quality related to industry requirements for production of devices having line widths from 5 to 0.18  $\mu\text{m}$ .

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.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

- D 1129 Terminology Relating to Water<sup>2</sup>
- D 1193 Specification for Reagent Water<sup>2</sup>
- D 1976 Test Method for Elements in Water by Inductively-Coupled Argon Plasma Atomic Emission Spectroscopy (ICP-AES)<sup>2</sup>
- D 2791 Test Method for Continuous Determination of Sodium in Water<sup>2</sup>
- D 3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry<sup>2</sup>
- D 4191 Test Method for Sodium in Water by Atomic Absorption Spectrophotometry<sup>2</sup>
- D 4192 Test Method for Potassium in Water by Atomic

- Absorption Spectrophotometry<sup>2</sup>
- D 4327 Test Method for Anions by Chemically Suppressed Ion Chromatography<sup>2</sup>
- D 4453 Practice for Handling of Ultra Pure Water Samples<sup>2</sup>
- D 4517 Test Method for Low-Level Total Silica in High-Purity Water by Flameless Atomic Absorption Spectroscopy (GFAAS)<sup>3</sup>
- D 4779 Test Method for Total, Organic or Inorganic Carbon in High-Purity Water by Ultraviolet (UV) or Both, or by High Temperature Combustion Followed by Gas Phase NDIR or Electrolytic Conductivity<sup>3</sup>
- 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 Electrolytic Conductivity<sup>2</sup>
- D 5391 Test Method for Electrical Conductivity and Resistivity of a Flowing High Purity Water Sample<sup>2</sup>
- D 5462 Test Method for On-Line Measurement of Dissolved Oxygen in Water<sup>2</sup>
- D 5542 Test Methods for Trace Anions in High Purity Water by Ion Chromatography<sup>2</sup>
- D 5544 Test Method for On-Line Measurement of Residue after Evaporation of High-Purity Water<sup>2</sup>
- D 5673 Test Method for Elements in Water by Inductively-Coupled Argon Plasma Mass Spectrometry (ICP-MS)<sup>2</sup>
- D 5996 Test Method for Measuring Anionic Contaminants in High-Purity Water by On-Line Ion Chromatography<sup>2</sup>
- D 5997 Test Method for On-Line Monitoring of Total Carbon, Inorganic Carbon, in Water by Ultraviolet, Persulfate Oxidation, and Membrane Conductivity Detection<sup>2</sup>
- 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 Pre-Sterilized Plastic Bag Method<sup>4</sup>

## 3. Terminology

3.1 *Definitions*— For definitions of terms used in this guide refer to Terminology D 1129.

<sup>1</sup> 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.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.02.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 10.04.

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *endotoxins, n*—substances or by-products usually produced by gram negative microorganisms that give a positive test for pyrogens, determined in accordance with the limulus lysate test (9.2).

3.2.2 *total bacterial counts, n*—total number of viable microorganisms present in the named sample, excluding anaerobic organisms, determined in accordance with Test Methods F 1094.

3.2.3 *total organic carbon (TOC), n*—carbon measured after inorganic carbon response has been eliminated by one of the prescribed ASTM test methods (such as elimination in accordance with Test Method D 4779).

3.2.4 *water, n*—water prepared in accordance with Specification D 1193, reagent grade Type I.

### 4. Significance and Use

4.1 This guide recommends the water quality required for the electronic and microelectronic industry. High purity water is required to prevent contamination of products during manufacture, which would otherwise result in an unacceptable, low yield of electronic devices.

4.2 The range of water purity is defined in accordance with the manufacturing process. The types of ultra pure water are defined with respect to device line width.

4.3 The limits on the impurities are related to current contamination specifications and to available analytical methods performed in a suitable, clean laboratory or using on-line methods. On-line and off-line methods are used in accordance with current industry practice. Concentration of the sample may be required to measure the impurities at the levels indicated in Table 1.

**TABLE 1 Requirements for Water Used in the Electronics and Semiconductor Industry**

Parameter	Type E-1	Type E-1.1	Type E-1.2	Type E-2	Type E-3	Type E-4
Linewidth (microns)	1.0–0.5	0.5–0.25	0.25–0.18	5.0–1.0	> 5.0	—
Resistivity, 25°C	18.2	18.2	18.2	17.5	12	0.5
Endotoxin unit (EU/mL)	0.03	0.03	0.03	0.25	—	—
TOC (µg/L)	5	2	1	50	300	1000
Dissolved oxygen (µg/L)	1	1	1	—	—	—
Residue after evaporation (µg/L)	1	0.5	0.1	—	—	—
SEM particles/L (micron range)						
0.1–0.2	1000	1000	200	—	—	—
0.2–0.5	500	500	100	3000	—	—
0.5–1	50	50	1	—	10 000	—
10	—	—	—	—	—	100 000
On-line particles/L (micron range)						
0.05–0.1	500	500	100	—	—	—
0.1–0.2	300	300	50	—	—	—
0.2–0.3	50	50	20	—	—	—
0.3–0.5	20	20	10	—	—	—
> 0.5	4	4	1	—	—	—
Bacteria/100 mL						
100 mL Sample	1	1	1	—	—	—
1 L Sample	1	1	0.1	10	10 000	100 000
Silica – total (µg/L)	3	0.5	0.5	10	50	1000
Silica – dissolved (µg/L)	1	0.1	0.05	—	—	—
Ions and metals (µg/L)						
Ammonium	0.1	0.10	0.05	—	—	—
Bromide	0.1	0.05	0.02	—	—	—
Chloride	0.1	0.05	0.02	1	10	1000
Fluoride	0.1	0.05	0.03	—	—	—
Nitrate	0.1	0.05	0.02	1	5	500
Nitrite	0.1	0.05	0.02	—	—	—
Phosphate	0.1	0.05	0.02	1	5	500
Sulfate	0.1	0.05	0.02	1	5	500
Aluminum	0.05	0.02	0.005	—	—	—
Barium	0.05	0.02	0.001	—	—	—
Boron	0.05	0.02	0.005	—	—	—
Calcium	0.05	0.02	0.002	—	—	—
Chromium	0.05	0.02	0.002	—	—	—
Copper	0.05	0.02	0.002	1	2	500
Iron	0.05	0.02	0.002	—	—	—
Lead	0.05	0.02	0.005	—	—	—
Lithium	0.05	0.02	0.003	—	—	—
Magnesium	0.05	0.02	0.002	—	—	—
Manganese	0.05	0.02	0.002	—	—	—
Nickel	0.05	0.02	0.002	1	2	500
Potassium	0.05	0.02	0.005	2	5	500
Sodium	0.05	0.02	0.005	1	5	1000
Strontium	0.05	0.02	0.001	—	—	—
Zinc	0.05	0.02	0.002	1	5	500

## 5. Classification

5.1 Six types of electronic grade water are described in this guide.

5.1.1 *Type E-1*—This water is classified as microelectronic water to be used in the production of devices having line widths between 0.5 and 1.0  $\mu\text{m}$ .

5.1.2 *Type E-1.1*—This water is classified as microelectronic water to be used in the production of devices having line widths between 0.25 and 0.5  $\mu\text{m}$ .

5.1.3 *Type E-1.2*—This water is classified as microelectronic water to be used in the production of devices having line widths between 0.18 and 0.25  $\mu\text{m}$ . It is the water of ultimate practical purity produced in large volumes and is intended for the most critical uses.

5.1.4 *Type E-2*—This water is classified as microelectronic water to be used in the production of devices that have dimensions between 1 and 5  $\mu\text{m}$ .

5.1.5 *Type E-3*—This grade of water is classified as macroelectronic water to be used in the production of devices having dimensions larger than 5  $\mu\text{m}$ . This grade may be used to produce larger components and some small components not affected by trace amounts of impurities.

5.1.6 *Type E-4*—This may be classified as electroplating water to be used in the preparation of plating solutions, the production of certain electronic grade chemicals, and other applications where the water being used is in constant contact with the atmosphere because of tank storage. This water purity is based upon the fact that the water may have had a significantly higher purity, but that it becomes contaminated because of tank storage and handling.

5.2 Components of the water system for producing electronic grade water shall be grouped into five general process sections for the purpose of simplifying the organization of the components of the systems. These processes are described in 5.2.1-5.2.5.

5.2.1 *Pretreatment*—The processes in this category include the addition of various types of coagulants, precipitating agents, clarifiers, sedimentation tanks, particulate filtration systems, including sand filters, disposable filter elements, ultrafilter membranes, and other particle removing systems. Adsorbent or entrapment beds may include greensand, activated carbon, and various synthetic materials specific for certain organic and inorganic impurities.

5.2.2 *Desalination*—This process is fundamental to the production of ultrapure water of all grades and may include more than one of the processes of ion exchange, distillation, reverse osmosis, electrodialysis, or continuous electrodeionization or all of the above. The size of the system governs the choice of the combination of desalination processes; for example the use of distillation in a laboratory that would not be practical in a large production plant. Various configurations of the different processes should be considered, including two-bed and mixed-bed demineralization, multi-stage reverse osmosis employing various types of membranes, and electrodialysis that might employ periodic polarity reversal techniques as well as the use of resins placed between the cation and anion membranes.

5.2.3 *Organic and Biological Removal Systems*—Removal of biological and organic contaminants is an important adjunct of any system used to prepare ultra pure water. Dissolved organic compounds can accumulate in the system during the process as well as being present in the original water. Methods of minimizing biological contamination include the addition of active chlorine (sodium hypochlorite), hydrogen peroxide, and ozone. Ultraviolet irradiation at 254 nm significantly reduces the growth of organisms. Ultraviolet irradiation at 185 nm produces traces of ozone that break down organic compounds to carbon dioxide. Adsorptive beds such as strong base anion resins as well as mixed resin beds may also be effective in removing biological contaminants. Membrane filters including reverse osmosis and ultrafilters may also remove biological impurities as well as organic molecules. Synthetic adsorbent columns ranging from porous resins to activated carbon may be effective in removing organics.

5.2.4 *Particulate Removal*—Particulate removal in the production of ultra pure water is differentiated from pretreatment to remove gross suspended substances. Particles of all types, biological, organic, or inorganic, significantly interfere with the production of electronic components. Processes used to remove particulate matter generally consist of the use of a microporous membrane structure of flat, cylindrical, or pleated configuration. In the case of ultrafilters and reverse osmosis units, they are of hollow fiber, tubular, or spiral wound. The choice of the particular membrane depends upon the pore size, characteristics of that membrane, and the size of particle to be removed. As a general rule, particles of a size larger than 10 % of the minimum dimension of the device being produced should be removed. The measurement of these particles becomes difficult at best and is generally dictated by the quality and pore size of the membrane used as the final filter.

5.2.5 *Storage and Distribution System*—This guide for the storage of electronic grade water during production is very important because impurities are added to the water in proportion to the solubility, area of contact, and time of contact between the water and materials of containment. Because it is important to minimize the contact with the materials of containment of storage, the volume of storage should also be minimized. Particular emphasis must be placed upon the atmosphere above the water that may contaminate the water with biological, organic, inorganic, and particulate impurities. The storage of E-1, E-1.1, E-1.2, E-2, and E-3 waters is impossible (if expecting to maintain purity); therefore, all storage should be before the final polishing, tertiary section.

5.2.5.1 The distribution systems also present a large area of contact between the water and pipe or tubing and, therefore, must be of a pure insoluble substance. Once again, the degree of contamination depends upon the solubility of the materials of contact as well as the time of contact. For that reason and because biological impurities tend to accumulate in stagnant water, the flow of water through the distribution system must be maintained on a continuous 24 hr basis. Contaminants probably dissolve in the water at a constant rate; therefore an increase in the velocity through the pipe will give an apparent

reduction in the contamination. However the flow rate, particularly through large pipes above 50 mm diameter that have a small surface to volume ratio, need not exceed 25 cm/s.

5.2.5.2 Plastic systems should be installed with care without the use of oils of any kind. Generally, cleaning prior to use is best accomplished by rinsing for several days with pure water. If the system has been idle for a period of time or was not clean when installed, a pre-rinse of nonionic detergent and 200 mg/L sodium hypochlorite might be required. The distribution outlets must also be of noncontaminating design and materials, with particular care given to minimize the possibility of back contamination of the system from the faucet or valve outlet.

### 5.3 Component Sections:

5.3.1 *Primary Section*—This section of the water system performing the primary purification of the input water should utilize one or more of the pretreatment and desalination processes noted in 5.2. The water produced in the primary section, should have a purity equivalent to Type E-4 and may or may not be stored prior to use or additional purification.

5.3.2 *Secondary Purification Section*—This section of the water system should be dedicated to upgrading the effluent of the primary section to the purity level of Type E-3 that will include organic and biological removal as well as additional desalination processes as necessary. Particulate removal may also be incorporated into this section of the purification train depending upon the processes used in the primary section. The water from the secondary purification section may be stored for use in a protected tank system or used directly from the purification train if a degradation of the resistance is to be avoided.

5.3.3 *Tertiary Section*—This section of the water system is dedicated to the removal of traces of ionized impurities, organics, and biological and particulate contaminants that will increase the purity to the requirements of Type E-1, E-1.1, E-1.2, or Type E-2. This section may include additional mixed bed ion exchange (regenerative or cartridge systems), ultraviolet light (the radiation of both 254 and 185 nm), and finally filtration, using the components described in 5.2.4. The water from this section of the system should be used without storage and should be recirculated continuously.

## 6. Requirements

6.1 Electronic grade water when referenced to this guide shall conform to the recommendations in Table 1. Additional recommendations, including the modification or elimination of certain test procedures, may be included as part of this guide by agreement between the parties concerned.

## 7. Reagents

### 7.1 Purity of Reagents:

7.1.1 *Types E-1, E-2, E-3, and E-4*—Reagent grade chemicals shall be used in all tests.

7.1.2 *Types E-1.1 and E-1.2*—Ultra pure chemicals shall be used in all tests. 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.

7.1.3 Unless otherwise indicated, all reagents shall conform to the specification of the Committee on Analytical Reagents of

the American Chemical Society<sup>5</sup>, where such specifications are available. 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.

### 7.2 Purity of Water:

7.2.1 *Types E-1, E-2, E-3, and E-4*—References to water shall be understood to mean Type I reagent grade water in accordance with Specification D 1193.

7.2.2 *Types E-1.1 and E-1.2*—Water equivalent to or better than E-1.2 is required.

## 8. Sampling

8.1 The test methods specified in Section 9 assume that great care and skill are required to obtain water samples to be tested. It is assumed that container and airborne contamination is minimized and operators should note possible sources of contamination due to the sampling procedure. It is recommended that the samples be handled as noted in Practice D 4453.

8.2 Extreme care must be exercised in handling samples for analyses. Pre-cleaned polystyrene flasks and high quality HDPE are recommended as sample containers. PFA or TFE-fluorocarbon will leach fluoride. Borosilicate glassware leach ions at low microgram per litre levels. The major contaminants from borosilicate glass are sodium (Na), potassium (K), boron (B), and silica (SiO<sub>2</sub>).

## 9. Test Methods

9.1 *Electrical Resistivity*—Test Method D 5391, Resistivity (conductivity) is measured off- or on-line depending on the application.

NOTE 1—18.25 MegOhm-centimeter is the theoretical limit for pure water at 25°C.

### 9.2 Endotoxins—Limulus Lysate Test<sup>5</sup>

9.3 *TOC*—Test Methods D 5173 and D 5997, involve on-line oxidation of organic materials and measurements of the carbon dioxide produced by conductivity or infrared photometry.

9.4 Dissolved oxygen should be measured on-line when required by Test Method D 5462.

### 9.5 Residue after evaporation uses Test Method D 5544.

9.6 *Particulate Matter (particles/L)*—Scanning electron microscopy (SEM) with an elemental analyzer accessory is useful for troubleshooting. On-line methods of data acquisition using laser technology are recommended for trend analysis.

9.7 *Bacteria (number of bacteria/100 mL)*—Test Methods F 1094, Cultures are run in triplicate using acceptable filters and sample sizes of 100 or 1000 mL. Total count of the media after incubation at 28°C for 48 to 72 hours depending on the

<sup>5</sup> *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

specifications. Epi-fluorescence microscopy has also been used to measure and identify bacteria.

9.8 *Silica*—Total silica ( $\mu\text{g/L}$ ) GFAAS, ICP-AES, and ICP-MS are suitable techniques, Test Method D 4517, D 1976, or D 5673. Dissolved silica ( $\mu\text{g/L}$  as  $\text{SiO}_2$ ) utilizes heteropoly blue photometry or ion chromatography for the lowest levels.

9.9 *Ionic Concentrations* ( $\mu\text{g/L}$ )—Many anions and cations may be determined by using ion chromatography either off-line by Test Method D 4327 or D 5542, or preferably on-line by Test Method 5996–96. Metals may be determined using ICP-AES (Test Method D 1976) or ICP-MS (Test Method D 5673) using suitable ICP conditions in accordance with the analytical requirement.

9.9.1 Copper—Practice D 3919.

9.9.2 Nickel—Practice D 3919.

9.9.3 Potassium—Test Method D 4192.

9.9.4 Sodium—Test Methods D 2791 and D 4191.

9.9.5 Zinc—Practice D 3919.

9.10 Where a test covering the particular impurity or contaminant is not sensitive enough to reliably measure the impurity at the level specified in Table 1, a suitable nonreferenced test shall be made by agreement between the concerned parties.

9.11 The sample may require concentration in order to measure the impurities at the levels indicated in Table 1.

## 10. Systems Operations

10.1 The size of the components of the primary, secondary, and tertiary sections of the water system is influenced by the ability of the user to recirculate all or some portion of the water after it has been used in a cleaning or component rinse operation. The term recirculation refers to the process of piping used water back to a point in the system where it may be purified or combined with water of approximately equal quality. From this point it is processed through the secondary section, and if necessary through portions of the primary section, followed by the tertiary section, and reused.

10.1.1 Water recovered from processes that impart contaminants to the water in large quantities, such as nonionic surfactants that may be difficult to remove because of the particular configuration of the system, should not be recirculated.

10.2 The quality of the effluent water depends upon the age, type, and method of regeneration of the ion exchange resins. Likewise, the flow rate through the ion exchange resin bed will affect the resistivity of the effluent water. The resin manufacturer's instructions relating to flow rate and regeneration procedure should be followed.

10.3 The use of microporous, reverse osmosis, or ultrafilter membranes as well as the use of plastic piping in the system may add a small amount of organic contamination to the water produced. Water users concerned with the most critical electronic device applications should be aware of this contamination and, where necessary, measure or monitor the level of organic contamination by means of a total organic carbon analysis, or other applicable analytical procedure.

10.3.1 Certain membranes contain small quantities of soluble impurities as a result of the manufacturing process. Care must be taken to avoid the use of the initial effluent from such membranes when membrane changes are made.

10.3.2 The periodic back flushing and cleaning required with the use of some types of ultra-filters or reverse-osmosis (RO) units imparts contaminants to the filter that the user must take care to flush from the membrane system before placing the system back on-line.

10.3.2.1 In certain applications involving the processing of sodium-sensitive, solid-state electronic devices, users may prefer to fast flush or back flush with a hydrogen peroxide or ozone solution to reduce the rinse time required when using the generally recommended sodium hypochlorite flush solution. The user should check with the manufacturer to make certain that the membranes resist the chemicals used and tolerate the type of flushing.

10.4 Because atmospheric gases and airborne impurities rapidly re-contaminate exposed water, in-line electrodes must be employed for determining the electrical resistivity of electronic grade waters of Types E-1, E-1.1, E-1.2, E-2, E-3, or E-4. Most pH measuring devices introduce impurities from electrodes to Types E-1, E-1.1 E-1.2, E-2, and E-3 waters that reduces the purity of the waters, thus making the pH measurement invalid.

## 11. Keywords

11.1 electronic; microelectronics; semiconductor; ultra pure water

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