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Standard Guide for
Ultra-Pure Water Used in the Electronics and Semiconductor Industries[1]

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1. Scope

1.1 This guide provides recommendations for water quality related to electronics and semiconductor-industry manufacturing. Six classifications of water are described, including water for line widths as low as 0.09 micron. In all cases, the recommendations are for water at the point of distribution (POD).

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

1.3 Users needing water qualities different from those described here should consult other water standards, such as Specification D 1193 and Guide D 5196.

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:* [2]

D 1129 Terminology Relating to Water
D 1193 Specification for Reagent Water
D 1976 Test Method for Elements in Water by Inductively-Coupled Argon Plasma Atomic Emission Spectroscopy
D 2791 Test Method for On-line Determination of Sodium in Water
D 3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry
D 4191 Test Method for Sodium in Water by Atomic Absorption Spectrophotometry
D 4192 Test Method for Potassium in Water by Atomic Absorption Spectrophotometry
D 4327 Test Method for Anions in Water by Chemically Suppressed Ion Chromatography
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
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 5196 Guide for Bio-Applications Grade Water
D 5391 Test Method for Electrical Conductivity and Resistivity of a Flowing High Purity Water Sample
D 5462 Test Method for On-Line Measurement of Low-Level Dissolved Oxygen in Water
D 5542 Test Methods for Trace Anions in High Purity Water by Ion Chromatography
D 5544 Test Method for On-Line Measurement of Residue After Evaporation of High-Purity Water
D 5673 Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry
D 5996 Test Method for Measuring Anionic Contaminants in High-Purity Water by On-Line Ion Chromatography
D 5997 Test Method for On-Line Monitoring of Total Carbon, Inorganic Carbon in Water by Ultraviolet, Persulfate Oxidation, and Membrane Conductivity Detection
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 of terms used in this guide refer to Terminology D 1129.

3.2 *Definitions of Terms Specific to This Standard:*

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[1] This guide is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.02 on General Specifications, Technical Resources, and Statistical Methods.

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[2] 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.

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

3.2.2 *total organic carbon (TOC)*, *n*—carbon measured after inorganic-carbon response has been eliminated by one of the prescribed ASTM test methods.

4. Significance and Use

4.1 This guide recommends the water quality required for the electronics and microelectronics industries. High-purity water is required to prevent contamination of products during manufacture, since contamination can lead to 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. In all cases, the water-quality recommendations apply at the point of distribution.

4.3 The limits on the impurities are related to current contamination specifications and to available analytical methods (either performed in a suitable clean laboratory or by on-line instrumentation). 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.

5. Classification

5.1 Six types of electronic-grade water are described in this guide. In all cases, the water-quality recommendations apply at the point of distribution.

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 µm.

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 0.35 µm.

TABLE 1 Requirements for Water at the Point of Distribution in the Electronics and Semiconductor Industries[A]

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	0.35–0.25	0.18–0.09	5.0–1.0	>5.0	—
Resistivity, 25°C (On-line)	18.1	18.2	18.2	16.5	12	0.5
TOC (µg/L) (on-line for <10 ppb)	5	2	1	50	300	1000
On-line dissolved oxygen (µg/L)	25	10	3	—	—	—
On-Line Residue after evaporation (µg/L)	1	0.5	0.1	—	—	—
On-line particles/L (micron range)						
0.05–0.1		1000	200	—	—	—
0.1–0.2	1000	350	<100	—	—	—
0.2–0.5	500	<100	<10	—	—	—
0.5-1.0	200	<50	<5	—	—	—
1.0	<100	<20	<1	—	—	—
SEM particles/L (micron range)						
0.1–0.2	1000	700	<250	—	—	—
0.2–0.5	500	400	<100	3000	—	—
0.5–1	100	50	<30	—	10 000	—
10	<50	<30	<10	—	—	100 000
Bacteria in CFU/Volume						
100 mL Sample	5	3	1	10	50	100
1 L Sample			10			
Silica – total (µg/L)	5	3	1	10	50	1000
Silica – dissolved (µg/L)	3	1	0.5	—	—	—
Anions and Ammonium by IC (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
Metals by ICP/MS (µg/L)						
Aluminum	0.05	0.02	0.005	—	—	—
Barium	0.05	0.02	0.001	—	—	—
Boron[B]	0.3	0.1	0.05	—	—	—
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

[A] The user should be advised that analytical data often are instrument dependent and technique dependent. Thus, the numbers in Table 1 are only guidelines.

[B] Boron is monitored only as an operational parameter for monitoring the ion-exchange beds.

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 0.09 and 0.18 µm. 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 µm.

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 µm. 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 grade may be classified as water used in preparation of plating solutions and for other applications where the water being used can be of lesser quality.

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, and 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 ultra-pure water of all grades, and may include more than one of the processes of ion exchange, reverse osmosis, electrodialysis, continuous electrodeionization, or all of the above. The size of the system governs the choice of the combination of desalination processes. 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, electrodeionization, and electrodialysis.

5.2.3 *Organic and Biological Removal Systems*—Removal of biological and organic contaminants is an important adjunct to 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 hydrogen peroxide and ozone. Ultraviolet irradiation at the 185 nm wavelength provides intense energy for breaking chemical bonds and produces traces of ozone. The 185 nm light lyses bacteria and breaks down organic compounds to organic acids and carbon dioxide. With the destruction of organics, TOC will be reduced. Therefore, 185 nm light should only be used upstream of the final ion-exchange component. Ultraviolet irradiation at 254 nm significantly reduces the growth of organisms by dislocating the DNA base pairs. This process prevents the bacteria from replicating. 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 that removes 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. The Final Filters are preceded by a bank of Prefilters with a 2× larger pore size. Ultrafilters and reverse-osmosis units may also be used for final filtration. The choice of the particular membrane depends in part upon the pore size, characteristics of that membrane, and the size of particle to be removed. As a general rule, particles should be removed if they are larger than 10 % of the minimum dimension of the device being produced.

5.2.5 *Storage and Distribution System*—The storage of electronic-grade water during production is very important, because impurities are added to the water in proportion to the solubility of the impurities, the area of contact, and time of contact between the water, the air, the materials of containment, or combinations thereof. Because it is important to minimize the contact of the water with the storage container, the volume of storage should also be minimized. A loop-distribution design is preferred to an antenna-distribution design, the latter of which can constitute dead legs during periods of non-use. Particular emphasis must be placed upon the atmosphere above the water, since those gases may contaminate the water with biological, organic, inorganic, and particulate impurities. High-purity nitrogen (≥99.999 % pure) should be used to blanket the atmosphere above the stored water. The storage of E-1, E-1.1, E-1.2, E-2, and E-3 waters is impossible (if expecting to maintain purity as described in Table 1); therefore, all storage should be before the tertiary section (see 5.3.3 for a discussion of "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 these reasons 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-h basis. The flow rate should be at least 3 ft/s, and 5 to 7 ft/s is recommended; this flow rate is particularly applicable through large pipes (that is, above 50 mm in diameter) that have a small surface-to-volume ratio. To maintain E-1, E-1.1, or E-1.2 specifications, a fluorinated-polymer piping such as PVDF is required.

5.2.5.2 Polymer material should be installed with care and 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 system sanitization using hydrogen peroxide or ozone might be required. The distribution outlets must also be of non-contaminating design and materials; particular care must be given to minimizing the possibility of back contamination of the system from the faucet or valve outlet. To maintain E-1, E-1.1, or E-1.2 specifications, a fluorinated-polymer piping such as PVDF is required.

5.3 *Component Sections*:

5.3.1 *Primary Section*—This section of the water system performs the primary purification of the input water. The primary section 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 bringing the effluent of the primary section up to the purity level of Type E-3. This section will include removal of organic and biological material, and will include 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 in a protected tank system, although the purity will not be maintained over time. If a degradation of the resistivity is to be avoided, the water should be used directly from the purification train.

5.3.3 *Tertiary Section*—This section of the water system is dedicated to the removal of traces of ionized impurities, organic materials, and biological and particulate contaminants. This removal process 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 (both 254 and 185 nm), and final filtration, using the components described in 5.2. The water from this section of the system should be used without storage and should be recirculated continuously through the final ion-exchange section.

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 among the parties concerned.

7. Reagents

7.1 *Purity of Reagents*:

7.1.1 Unless otherwise indicated, all reagents shall conform to the specification of the Committee on Analytical Reagents of the American Chemical Society[3] where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is sufficiently pure to permit its use without lessening the accuracy of the determination.

7.1.2 *Types E-1, E-2, E-3, and E-4*—Reagent-grade chemicals[3] should be used in all tests.

7.1.3 *Types E-1.1 and E-1.2*—The purest chemicals available should be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is sufficiently pure to permit its use without lessening the accuracy of the determination.

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; operators should note possible sources of contamination that are 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. Bottle materials that are acceptable for one analysis may or may not be acceptable for another. For very high-purity water, bottles should be analytically prequalified before use. PFA or TFE-fluorocarbon will leach fluoride. Borosilicate glass leaches ions at low microgram-per-liter levels. The major contaminants from borosilicate glass are sodium (Na), potassium (K), boron (B), and silica (SiO_2).

9. Test Methods

NOTE 1—The user should be advised that analytical data often are instrument dependent or technique dependent.

9.1 *Electrical Resistivity*—Test Method D 5391 measures resistivity on-line.

9.2 *TOC*—Test Methods D 5173 and D 5997 involve online oxidation of organic materials and measurements (by conductivity or infrared photometry) of the carbon dioxide produced. Water at less than 10 ppb levels should be analyzed by on-line instrumentation for greatest accuracy.

9.3 *Dissolved Oxygen*—Test Method D 5462 measures dissolved oxygen on-line.

9.4 *On-line Residue*—Test Method D 5544 measures online residue after evaporation.

9.5 *Particulate Matter* (particles/L):

9.5.1 On-line methods using laser technology are recommended for real-time trend analysis. The alternative of scanning electron microscopy (SEM) with an elemental-analyzer accessory is useful for particle counting and troubleshooting.

9.5.2 Most particle counters are actually particle monitors. These instruments are most useful for monitoring change, rather than providing an absolute number. Caution should be used in quantitating particles near the instrument's background level. Particle counters are calibrated from surrogate or natural materials and provide a reference in size to such calibration materials. Depending on the size of the particles to be monitored, different particle-counting techniques can be useful for the monitoring of process performance and for troubleshooting. These techniques include light obscuration, electrozone detection, or forward-scatter particle-counting methods. These techniques provide size and count information in a sample, but the information is referenced to the type of calibration materials used.

9.5.3 Laser turbidimeters are also available for use in particulate detection. These techniques are sensitive to particles down in the nm diameters. Such techniques are best used

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[3] *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.

for the monitoring of a breakthrough event, and do not provide descriptive information.

9.6 *Bacteria by Culture* (Colony Forming Units (CFU)/ Volume)—Test Methods F 1094 provides for running cultures (in triplicate) by concentrating samples on acceptable filters; sample sizes are 100 mL, 1000 mL or larger. Total count of the media is conducted after incubation. Incubation temperature ranges from 25°C to 28°C and incubation time ranges from 48 hours to 7 days; choice of both parameters depend on the within-company specifications. The common industry standard is 28 ± 2°C for 48 hours.

9.7 *Silica*:

9.7.1 *Total Silica* (µg/L)—Test Methods D 4517, D 1976, and D 5673 use GFAAS, ICP-AES, and ICP-MS, respectively; all three of these techniques are suitable.

9.7.2 *Dissolved Silica* (µg/L as SiO_2)—Methods for dissolved silica utilize heteropoly-blue photometry or ion chromatography for the lowest levels.

9.8 *Ionic Concentrations* (µg/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 D 5996.

9.9 *Metals* (µg/L)—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 requirements. Currently, ICP-MS equipment with lower detection levels is available; examples are sector field, high-resolution ICP-MS (HR-ICP-MS), quadrupole-ICP-MS (A-ICP-MS) fitted with a Collision Cell (CCT) or Dynamic Reaction Cell (DRC), or combinations thereof.

9.9.1 *Copper, Nickel, and Zinc*—Practice D 3919.

9.9.2 *Potassium*—Test Method D 4192.

9.9.3 *Sodium*—Test Methods D 2791 and D 4191.

9.10 Where a referenced test is not sensitive enough to measure the impurity reliably at the level specified in Table 1, a suitable non-referenced test shall be used by agreement among the concerned parties.

9.11 The sample may need to be concentrated in order to measure the impurities at the levels indicated in Table 1.

10. Recycle Water

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 recycle all or some portion of the water after it has been used in a cleaning or component-rinse operation. The term "recycle" refers to the process of piping used water back to where it may be purified or combined with water of approximately equal quality. From this point, the water 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 process streams that impart contaminants to the water in large quantities should not be reintroduced to the UPW system without additional comprehensive pretreatment steps.

11. Keywords

11.1 electronic; microelectronics; semiconductor; ultra-pure water

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