

SEMI F63-0918

GUIDE FOR ULTRAPURE WATER USED IN SEMICONDUCTOR PROCESSING

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1 Purpose

1.1 This Guide may be used:

- To establish performance criteria when purchasing ultrapure water (UPW) purification equipment.
- To set the process control parameters for UPW-system operation.
- To establish quality expectations for the supplied UPW.

NOTE 1: These suggested guidelines are published as technical information and are intended for informational purposes only.

2 Scope

2.1 UPW is used extensively in the production of semiconductor devices for all wet-processing steps (including wafer rinsing). UPW purity is therefore critical to the manufacture of semiconductors. This Guide provides UPW quality parameters and background information for the decision-making process related to new or retrofit facilities that manufacture semiconductors with line widths of 32 nm and smaller.

2.2 The goal of members of the International Roadmap for Devices and Systems (IRDS) UPW committee is to look years ahead on a continual basis to assess the semiconductor industry's future UPW technology requirements. In addition to IRDS input, the SEMI committee contributed its knowledge of the feasibility and cost viability of the technical solutions required to support UPW quality values specified in this Guide.

2.3 The information in this Guide has been developed from the following sources:

- IRDS risk assessment with input from facility- and manufacturing-experts and technology providers in the semiconductor manufacturing industry.
- The results of UPW testing at semiconductor manufacturing sites as measured by independent laboratories that test high purity water for the semiconductor industry.
- Specifications from water-system equipment manufacturers.
- Input from UPW producers and users at SEMI Standards committee meetings, and also through the balloting process.

NOTICE: SEMI Standards and Safety Guidelines do not purport to address all safety issues associated with their use. It is the responsibility of the users of the Documents to establish appropriate safety and health practices, and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 This Guide is not intended to define UPW system performance requirements. It only provides a reference set of parameters and their values, considered to be satisfactory for the advanced semiconductor manufacturing needs. In some cases, the values are limited by the feasibility of the available treatment technology or metrology. It is expected that semiconductor company experts will define the performance requirements for the UPW system based on specific manufacturing process sensitivities, with reference to the information provided in this Guide.

3.2 The purity of water generated in water systems differs. These guidelines were developed for properly maintained, state-of-the-art UPW systems as found in high-end semiconductor manufacturing plants and may not reflect the needs of lesser demanding end-users.

3.3 This Guide is not intended to be used to define the criteria needed for selecting process tools.

3.4 This Guide defines parameters that apply to the quality of UPW required in semiconductor processing when proper analytical hookup and tool installation is made.

3.5 This Guide may include suggested values that are at the detection limit of today's available, cost-viable, analytical instrumentation or techniques. UPW system owners, engineers, and technicians are expected to decide whether the benefits of meeting the suggested values outweigh the associated time and costs. The quality of the data measured will depend on which testing method and calibration techniques are used. Laboratory or metrology vendors may be requested to provide additional analytical validation data to confirm detection limits before internal specification values are set.

4 Referenced Standards and Documents

4.1 SEMI Standards and Safety Guidelines

SEMI C1 — Guide for the Analysis of Liquid Chemicals

SEMI C10 — Guide for the Determination of Method Detection Limits

SEMI C79 — Guide to Evaluate the Efficacy of Sub-15 nm Filters Used in Ultrapure Water (UPW) Distribution Systems

SEMI C93 — Guide for Determining the Quality of Ion Exchange Resin Used in Polish Applications of Ultrapure Water System

SEMI F61 — Guide to Design and Operation of a Semiconductor Ultrapure Water System

4.2 ASTM Standards¹

ASTM D859 — Standard Test Method for Silica in Water

ASTM D4327 — Standard Test Method for Anions in Water by Chemically Suppressed Ion Chromatography

ASTM D4517 — Standard Test Method for Low-Level Total Silica in High-Purity Water by Flameless Atomic Absorption Spectroscopy

ASTM D5127 — Standard Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industries

ASTM D5173 — Standard Guide for On-Line Monitoring of Total Organic Carbon in Water by Oxidation and Detection of Resulting Carbon Dioxide

ASTM D5391 — Standard Test Method for Electrical Conductivity and Resistivity of a Flowing High Purity Water Sample

ASTM D5544 — Standard Test Method for On-Line Measurement of Residue After Evaporation of High-Purity Water

ASTM D6317 — Standard Test Method for Low Level Determination of Total Carbon, Inorganic Carbon and Organic Carbon in Water by Ultraviolet, Persulfate Oxidation, and Membrane Conductivity Detection

ASTM D7126 — Standard Test Method for On-Line Colorimetric Measurement of Silica

ASTM F1094 — Standard 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

4.3 Other Documents

International Roadmap for Devices and Systems (IRDS)²

Huber S. A. Characterization of Aquatic Humic and non-Humic Matter with Size-Exclusion Chromatography - Organic Carbon Detection - Organic Nitrogen Detection (LC-OCD-OND). Water Research, v. 45 (2011): pp 879–885.

¹ ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: 610.832.9585, Fax: 610.832.9555, <http://www.astm.org>

² <http://www.IRDS.net>

Libman V. Blackford D., Wilcox D., Burkhart M. UPW ITRS and SEMI Committee Progress in Developing Guidelines for Advanced Semiconductor Manufacturing, ULTRAPURE WATER Conference, Portland, OR, 2013.

Guidelines for the Application of SEM/EDX Analytical Techniques to Particulate Matter Samples. EPA#600/R-02/070.

Zerfas B., Sullivan L., and Libman V. Bacteria in Ultrapure Water: Myth Versus Reality, Part 2. ULTRAPURE WATER Conference, Portland, OR, 2017.

APHA Method 9215 (Heterotrophic Plate Count): Standard Methods for the Examination of Water and Wastewater. American Public Health Association. Code of Federal Regulations: 40 CFR 141.121

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 General terms and acronyms used in this standard are listed and defined in SEMI F61.

5.2 Abbreviations and Acronyms

5.2.1 *EDS* — energy dispersive X-ray spectroscopy

5.2.2 *LPC* — laser particle counter

5.2.3 *POC* — point of connection

5.2.4 *POD* — point of distribution

5.2.5 *POE* — point of entry

5.2.6 *POM* — point of measurement

5.2.7 *POP* — point of process

5.2.8 *POU* — point of use

5.2.9 *SEM* — scanning electron microscopy

5.3 Definitions

5.3.1 *points of measurement (POM)* — of the specified parameters follow commonly accepted terminology in semiconductor industry (UPW ITRS 2015, Table YE1):

5.3.1.1 *point of connection (POC)* — is at Outlet of lateral take off valve.

5.3.1.2 *point of distribution (POD)* — is at Outlet of final filtration in UPW system.

5.3.1.3 *point of entry (POE)* — is at Inlet of wet bench or sub-equipment.

5.3.1.4 *point of process (POP)* — is at Wafer in production.

5.3.1.5 *point of use (POU)* — is at Inlet of wet bench bath, spray nozzle, or connection point to piping, which is also used for other chemicals.

6 Use of the Guidelines

6.1 Supplemental information, including recommendations on how to use this Document, is provided at the end of this Guide. The supplemental information is critical to understanding the intent of this Guide.

6.2 The supplemental information should be considered when making decisions on UPW performance requirements. Any additional data about manufacturing process sensitivities should also be considered when making a final decision about appropriate UPW quality.

7 Units

7.1 Parts per million (ppm) is equivalent to $\mu\text{g/mL}$ or mg/L , where 1 L approximately equals 1 kg.

7.2 Parts per billion (ppb) is equivalent to ng/mL or $\mu\text{g/L}$, where 1 L approximately equals 1 kg.

7.3 Parts per trillion (ppt) is equivalent to pg/mL or ng/L, where 1 L approximately equals 1 kg.

7.4 Micrometer (μ) is a unit of length equal to one millionth of a meter, or one thousandth of a millimeter.

7.5 Colony forming units (CFU) is a measurement of bacteria organisms as referred to in ASTM F1094.

8 Description of Parameter Tests

NOTE 2: SEMI Guides do not require analytical data to support analytical methods, therefore the recommendations of specific analytical methods are only for informational purposes. Alternative methods may also be applicable (see SEMI C1 for analytical validation methodology).

8.1 Resistivity (*megohm-centimeters or $M\Omega\cdot cm$*)

8.1.1 Resistivity (reciprocal of electrical conductivity) is measured accurately with on-line instrumentation only. 18.18 $M\Omega\cdot cm$ is the theoretical upper limit for pure water at 25°C.

8.2 Temperature

8.2.1 Temperature stability (K) is defined as a range of the temperature deviation from the facility-specified temperature target. The temperature is measured with an online sensor installed inline.

8.2.2 Temperature gradient (K per interval) is defined as the rate of change of temperature within a specific interval of time.

8.3 Nonvolatile Residue (NVR)

8.3.1 NVR or a residue after evaporation (usually expressed in units of ppt) is measured using an online, NVR monitor.

8.4 Total Organic Carbon (TOC) (*usually expressed in units of ppb*) also known as Total Oxidizable Carbon

8.4.1 Conductivity cells or infrared photometry can be used to measure the carbon dioxide created by the oxidation of organic materials.

8.5 Dissolved Oxygen (*usually expressed in units of ppb*)

8.5.1 Dissolved oxygen is measured accurately with only online instrumentation.

8.6 Particulate Matter (*Particles/L*)

8.6.1 Currently, accurate trend analysis requires online methods using particle counters. Since no method is currently available (although several promising methods are under development) for accurately monitoring particles at the target size (see Table 1), no specific recommendation for the metrology is provided (see also additional clarification in § 10).

8.7 Bacteria (*CFU/L*)

8.7.1 Triplicate samples are cultured based on ASTM F1094.

8.8 Silica

8.8.1 Total Silica (usually expressed in units of ppb) is measured by graphite furnace atomic absorption spectrophotometry (GFAAS), inductively-coupled plasma atomic emission spectroscopy (ICP-AES), or high resolution-inductively coupled plasma-mass spectroscopy (HR-ICP-MS) or by digestion with Heteropoly Blue Photometry using ASTM D7126.

8.8.2 Dissolved silica (usually expressed in units of ppb as SiO_2) is measured by Heteropoly Blue Photometry, or by ion chromatography.

8.9 Ions and Metals (*usually expressed in units of ppt*)

8.9.1 Anions and cations such as ammonium, bromide, chloride, fluoride, nitrate, nitrite, phosphate, and sulfate, can be detected using ion chromatography.

8.9.2 The 26 elements included in this Guide are listed in Table 1.

NOTE 3: Up to 68 elements, including the 26 of interest can be determined by GFAAS, ICP-AES, or ICP-MS.

9 Recommended Ultrapure Water Quality

9.1 Table 1 lists each parameter with its range of performance.

9.2 Table 1 should be considered together with the supplemental information (see § 10).

Table 1 Recommended Ultrapure Water Quality

PARAMETER	Point of Measurement of the Specified Parameters	Typical Linewidth <0.045 MICRONS	
		PERFORMANCE	Method Detection Limit (MDL) ^{#1}
Online Particles (#/mL) ^{#3} , see § 10.7	Feed to the Final Filters	<1000 (<100) ^{#5}	TBC ^{#10}
SEM off-line Particles, (#/L) @ 50 nm and larger ^{#7}	POD	<300	<100
Silica			
Silica – Total (ppb), § 10.9	Feed to the Final Filters	<0.05	<0.05
Silica – Dissolved or Reactive (ppb as SiO ₂), § 10.9	Feed to the Final Filters	<0.05	<0.05
Online TOC (ppb), see § 10.3	POD	<1	0.05
Online Dissolved Oxygen (ppb), see § 10.4	POD	<10	±0.2
On-Line Dissolved Nitrogen (ppm), for megasonic cleans only, see § 10.5	POP	8 to 18	±0.3
Dissolved Nitrogen Stability (ppm), for megasonic cleans only, § 10.5	POP	±2	±0.3
Residue After Evaporation Online (ppt), see § 10.5	POD	<100	<20
Hydrogen Peroxide (ppb), see § 10.13	POD	10	10
Bacteria (CFU/100 mL), see § 10.8	POD		
100 mL Sample	POD	<1	1
Ions (ppt)			
Ammonium	POD	<50	10
Bromide	POD	<50	10
Chloride	POD	<50	5
Fluoride	POD	<50	5
Nitrate	POD	<50	5
Nitrite	POD	<50	5
Phosphate	POD	<20	10
Sulfate	POD	<50	10
Metals and B (ppt)			
Aluminum	POD	<1	0.5
Antimony	POD	<1	0.5
Arsenic	POD	<1	0.5
Barium	POD	<1	0.5
Boron	POD	<50	15
Cadmium	POD	<1	0.5
Calcium	POD	<1	0.5

PARAMETER	Point of Measurement of the Specified Parameters	Typical Linewidth <0.045 MICRONS	
		PERFORMANCE	Method Detection Limit (MDL) ^{#1}
Chromium	POD	<1	0.5
Cobalt	POD	<1	0.5
Copper	POD	<1	0.5
Iron	POD	<1	0.5
Lead	POD	<1	0.5
Lithium	POD	<1	0.5
Magnesium	POD	<1	0.5
Manganese	POD	<1	0.5
Molybdenum	POD	<1	0.5
Nickel	POD	<3	1.6
Potassium	POD	<1	0.5
Platinum*	POD	<1	0.5
Sodium	POD	<1	0.5
Strontium	POD	<1	0.5
Tin	POD	<1	0.5
Tungsten	POD	<1	0.5
Titanium	POD	<1	0.5
Vanadium	POD	<1	0.5
Zinc	POD	<1	0.5
Resistivity Online @ 25°C (MΩ·cm), see § 10.1	POD	not less than 18.18	±0.20
Temperature Stability (K) (for immersion litho), see § 10.2	POP	±1	Not Applicable ^{#2}
Temperature Gradient (K/10 min.) (for immersion litho), see § 10.2	POP	<0.1	Not Applicable ^{#2}
Pressure , see § 10.12	Consideration Only		

#1 Method Detection Limits (MDL) as reported by the analytical vendors.

#2 The specified value is relative to the absolute temperature target chosen by a semiconductor manufacturing facility and therefore the accuracy or detection limits are 'not applicable.' The sensor suppliers state that temperature stability is within 0.1% of the reading for >2 years' operation, which is well within the expectations implied by the specified values.

#3 Particles size is specific to the dimensions of the manufacturing node (see § 10 for additional clarifications). The IRDS definition of the 'killer' particle size uses the roadmap's definition of 50% Logic ½ Pitch (nm).

#4 See § 10.8 for additional clarification.

#5 The killer size of the particles depends on the manufacturing technology generation (process sensitivity) in the fab. Per UPW IRDS the concentration of the particles in UPW does not depend on the killer particle size. The target value of the killer particles upstream to the final filters is defined based on the following assumptions:

#6 Target killer particle concentration in UPW is 10/mL per UPW IRDS.

#7 Best-in-class filters are used providing minimum of 99% removal efficiency for the killer particles and this is confirmed by SEMI C79 test. Tighter limit may be required for lower retention efficiency.

#8 Power law approximation is used for particles monitoring using existing online particle counters (see § 10.14). Power-law coefficient of 3 is recommended.

#9 Value in parenthesis is the limit specifically for EUV mask facility, which is particularly critical for particles control.

#10 TBC = To Be Confirmed. The actual detection limit depends on the type of the on-line particle counting instrument used. The detection limit should be at least an order of magnitude better than the target level of the particles to be controlled.

#11 The Method for SEM particle analysis is based on EPA#600/R-02/070. The filter pore size should be 50 nm with a minimum sample volume of 500 L (1000 L recommended). Refer to § 10.7 for additional details.

#12 The metal target values are based on the current UPW IRDS recommendations. The MDL for metals was provided for reference by a representative advanced material laboratory, using High Resolution ICP-MS for analysis without special sample pre-concentration. MDL for nickel