Background Statement for SEMI Draft Document 6086
Revision to SEMI F75-1102 (Reapproved 0309):
GUIDE FOR QUALITY MONITORING OF ULTRAPURE WATER USED IN SEMICONDUCTOR MANUFACTURING

NOTICE: This Background Statement is not part of the balloted item. It is provided solely to assist the recipient in reaching an informed decision based on the rationale of the activity that preceded the creation of this ballot.

NOTICE: For each Reject Vote, the Voter shall provide text or other supportive material indicating the reason(s) for disapproval (i.e., Negative[s]), referenced to the applicable section(s) and/or paragraph(s), to accompany the vote.

NOTICE: Recipients of this ballot are invited to submit, with their Comments, notification of any relevant patented technology or copyrighted items of which they are aware and to provide supporting documentation. In this context, 'patented technology' is defined as technology for which a patent has been issued or has been applied for. In the latter case, only publicly available information on the contents of the patent application is to be provided.

Background

This document is a complete rewrite of the existing SEMI F75 ultrapure water guide. The objective is to provide minimum reference information and guidance to assist the end user with developing specifications for cost effective and reliable UPW system monitoring program. This document is submitted for ballot together with the revision of SEMI F61, guide for UPW design and operation, and it is recommended to be used in conjunction with it.

Since this document is a guide it is not intended to tell the reader how exactly UPW system monitoring should be defined, rather it suggests what type of instruments and analytical methods are important as well as providing reference information based on the current industry experience (contributed by the team of experts involved in the document development, who represented perspectives of the end users, equipment/material suppliers, analytical experts, and consultants).

This document is also intended to continue to be updated every two years, keeping it in alignment with the industry needs as defined by UPW IRDS. This document is based on current SEMI F63 UPW Quality Guide and references other relevant SEMI UPW related documents, such as SEMI F57 and numerous ASTM standards commonly used for UPW monitoring.

The ballot results will be reviewed and adjudicated at the meetings indicated in the table below. Check www.semi.org/standards under Standards Calendar for the latest update.

Review and Adjudication Information

	Task Force Review	**Committee Adjudication**
Group:	Ultra Pure Water TF	NA Liquid Chemicals Committee
Date:	Monday, April 3, 2017	Tuesday, April 4, 2017
Time & Time zone:	8:00 AM to 11:00 AM, Pacific Time	3:00 PM to 6:00 PM, Pacific Time
Location:	SEMI Headquarters	SEMI Headquarters
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This meeting's details are subject to change, and additional review sessions may be scheduled if necessary. Contact the task force leaders or Standards staff for confirmation. Telephone and web information will be distributed to interested parties as the meeting date approaches. If you will not be able to attend these meetings in person but would like to participate by telephone/web, please contact Standards staff.

NOTICE: This Document was completely rewritten in 2017.

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SEMI Draft Document 6086
Revision to SEMI F75-1102 (Reapproved 0309):
GUIDE FOR QUALITY MONITORING OF ULTRAPURE WATER USED IN SEMICONDUCTOR MANUFACTURING

This standard was technically approved by the global Liquid Chemicals Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on [date TBD]. It was available at www.semi.org in XXXX. Originally published November 2002.

NOTICE: This Document was completely rewritten in 2017.

1 Purpose

1.1 This Guide describes potential sources of contaminants, their impact on semiconductor manufacturing and available options for monitoring these contaminants. This Guide should be used in conjunction with SEMI F61 and SEMI F63. Together these Guides provide recommendations for facility engineers and other manufacturing and quality professionals who are responsible for establishing programs to monitor and control the quality of their ultrapure water (UPW) systems, through to point of use (POU). These Guides may be used to help determine the parameters that should be monitored for UPW that is produced, distributed and used throughout the manufacturing facility, and the frequency and location of testing.

NOTE 1: These suggested guides 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. Ultrapure water systems need to be tested and monitored to ensure that the UPW being produced matches the specifications established by the manufacturing process. The purity of the UPW may affect device yield unless a wide range of parameters are closely controlled at the point of distribution (POD). Semiconductor devices are currently being designed with smaller linewidths (<65 nm) and are therefore more susceptible to low level impurities.

2.2 UPW systems are monitored for continuous performance for desired and achievable levels of quality. Action limits are generally set to determine when system performance data warrants that corrective action is needed. Table 1, Parameters and Range of Performance, in SEMI F63 may be a useful reference for establishing quality levels.

2.3 In more critical processes, the quality of the UPW also needs to be monitored at the POU where the UPW is in contact with the wafer. The quality of the UPW should not be expected to be identical to the quality of the UPW being produced at final filter (FF), which is not subject to conditions within the tool or distribution system.

2.4 This Guide (SEMI F75) is the third in a series of SEMI Guides developed for UPW which include SEMI F61, a Standard defining the performance of a UPW system, and SEMI F63, a Standard defining the quality of UPW.

2.5 This Guide provides information about the frequency and location of sampling for those parameters that are not available from online analyzers. Frequency of sampling should be based on the specifications set by manufacturing for the quality of the POD UPW, the number and locations of online analyzers, the stability of the incoming feed water to the system, and the historical performance of the UPW system over time.

2.6 The SEMI Guides may also be used to establish process control criteria for the incoming feedwater, performance of UPW system components and POU rinse baths.

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.

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3 Limitations

3.1 The SEMI Guides have been developed with consideration of various other relevant sources but they are not intended to be identical to, or consistent with, any other industry document or standard.

3.2 The SEMI Guides do not specifically address monitoring and testing of recycled or reclaimed water systems; additional test parameters, such as quantification and identification of organic species in reclaim water, should be added to monitoring programs for reclaim and reuse waters.

3.3 Measuring the purity of UPW can be challenging. Many online instruments (such as sodium analyzers, TOC analyzers, silica analyzers, optical particle counters, and nonvolatile residue analyzers) provide very low limits of detection but may not be capable of being calibrated in the range of detection or may have very poor accuracy at low levels therefore on-line analyzers should primarily be used for trend analysis. Many tests can still be performed more accurately and reproducibly in a laboratory environment but taking batch samples can prove time-consuming and be prone to sampling error.

3.4 To maintain their relevance, SEMI Documents must be updated on a regular basis. This Guide, SEMI F61 and SEMI F63 should all be updated at the same time in order to ensure continuity for the users of all three Documents. All three Documents will be updated on a two-year cycle.

4 Referenced Standards and Documents

4.1 *SEMI Standards and Safety Guidelines*

SEMI F57 — Specification for Polymer Materials and Components Used in Ultrapure Water and Liquid Chemical Distribution Systems

SEMI F61 — Guide for Ultrapure Water System Used in Semiconductor Processing

SEMI F63 — Guide for Ultrapure Water Used in Semiconductor Processing

4.2 *ASTM Standards*[1]

ASTM D859 — Standard Test Method for Silica in Water

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

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

ASTM D5173 — Standard 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

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

ASTM D5462 — Standard Test Method for On-Line Measurement of Low-Level Dissolved Oxygen in WaterASTM 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 — Test Method 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

4.3 *Other Documents*

ISO/IEC 17025:2005 — General Requirements for the Competence of Resting and Calibration Laboratories

Libman S. and Huber S. “An Overview of LC-OCD – Organic Speciation For Critical Analytical Tasks In the Semiconductor Industry.” *Ultrapure Water Journal*, Volume 31, Number 3, May/June 2014, pp. 10-16

[1] ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: +1.610.832.9585, Fax: +1.610.832.9555, http://www.astm.org

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

5 Terminology

5.1 *Abbreviations and Acronyms*

NOTE 2: All other abbreviations and acronyms are defined in the SEMI International Standards: Compilation of Terms available at Web site, http://www.semi.org/.

5.1.1 *CFU* — colony-forming units

5.1.2 *DO* — dissolved oxygen

5.1.3 *EDI* — electro-deionization

5.1.4 *EDX* — energy dispersive x-ray microanalysis

5.1.5 *FF* — final filter

5.1.6 *IC* — ion chromatography

5.1.7 *NDIR* — non-dispersive infrared analysis

5.1.8 *OPC* — optical particle counters

5.1.9 *POD* — point of distribution

5.1.10 *POU* — point of use

5.1.11 *RO* — reverse osmosis

5.1.12 *TDS* — total dissolved solids

5.1.13 *THM* — trihalomethanes

5.1.14 *LC*-OCD — liquide chromatography with organic carbon detector

6 Units

6.1 Parts per million (ppm) is equivalent to µg/mL or mg/L.

6.2 Parts per billion (ppb) is equivalent to ng/mL or µg/L.

6.3 Parts per trillion (ppt) is equivalent to pg/mL or ng/L.

7 Use of the Guides

7.1 Monitoring programs should reflect the age and complexity of the UPW equipment and the needs of the manufacturing process.

7.2 The quality of UPW at the POU may be negatively impacted by contamination sources within a tool, the design of the tool, the materials of construction of the tool and piping distribution, and contamination loading in the bath from build-up after multiple rinse cycles.

7.3 Sampling methods and contamination control are of paramount importance when attempting to measure the listed parameters at very low levels of sensitivity.

7.4 The quality of the data measured may depend on which testing methods and calibration techniques are used. Consequently, trends observed in the values may be more meaningful than absolute values, especially for certain online monitors calibrated for ultrasensitive detection. In addition, online and off-line measurements may not correlate depending on the measurement technique and level of sensitivity of the measurement.

8 Tests for Monitoring UPW Production and Distribution

NOTE 3: Since SEMI Guides do not require analytical data or methods to support them, the recommendations of specific analytical methods are only for informational purposes. Alternative methods may also be applicable.

NOTE 4: See Table 1, § 9, for a summary of recommended testing frequency and sampling points.

8.1 *Resistivity (megohm-centimeters [mΩ·cm])*

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8.1.1 Resistivity (the inverse of conductivity and a general measure of ionic activity) is measured by an online meter. The resistivity of UPW should be approximately 18.2 mΩ·cm depending on the resolution of the instrument.

NOTE 5: 18.18 MΩ·cm is the theoretical upper limit for pure water at 25°C.

8.2 *Total Oxidizable (Organic) Carbon (TOC) (ppb)*

8.2.1 *Carbon Sources in Water Supplies*

8.2.1.1 Incoming feed water contains both inorganic and organic carbon. Inorganic carbon as dissolved carbon dioxide (CO_2), bicarbonate, and carbonate can be present at high ppm levels according to the geology of the water supply. Organic carbon includes natural organic matter (NOM) input and man-made (synthetic) contaminants such as pesticides and fertilizers. Ground waters normally have significantly lower organic content than surface waters. To remove the majority of organics, most UPW systems employ reverse osmosis, anion exchange resin, and ultraviolet (UV) destruction. Some volatile organics, such as trihalomethanes (THM), may be reduced by the use of vacuum degasification. The control of organics is essential for preventing organic fouling and maintaining treatment at high efficiency. In addition, reduced organic matter will limit the nutrients available for bacteria growth. Increasing TOC values at the POD may be due to deterioration of the incoming water quality, degradation of system components, contamination from routine operational maintenance, or return contamination from the factory.

8.2.2 *Method of TOC Measurement for Source Water*

8.2.2.1 There are various methods for measuring TOC and several TOC analyzers are available. TOC measurement generally involves the oxidation of organic materials by means of temperature, UV radiation, and/or chemicals. The CO_2 produced by these reactions can be measured by non-dispersive infrared analysis (NDIR) or conductivity (resistivity) differential. The exact method used varies depending upon the TOC instrumentation employed. Different methods have different recoveries of various organics and therefore affect the TOC readings. Some instruments require the use of a carrier gas such as air or nitrogen while others measure TOC directly.

8.2.2.2 Special organic speciation may be required to identify the sources of the contamination and define adequate measures for correcting TOC levels. Liquide chromatography with organic carbon detector (LC-OCD) is recommended for such troubleshooting. This method can be used for both high ppm and low ppb levels of TOC speciation.

8.2.3 *Monitoring TOC in the UPW System*

8.2.3.1 Refer to ASTM D5173 and ASTM D6317 for TOC analysis in UPW.

8.2.3.2 TOC is a useful test to measure the organic removal effectiveness of the UPW system components including carbon, reverse osmosis (RO), degasification, and ion exchange. After the RO, TOC typically drops from low ppm levels in the source water to mid ppb range, and to single digit ppb levels after the mixed resin beds and UV TOC reduction units.

8.2.3.3 TOC may be measured at the point-of-use to determine quality changes from the distribution system and the manufacturing tool. Short wavelength UV (185 nm) is capable of breaking up residual organics into charged organic molecules. TOC that survives to the point of use in a UPW system is typically either ‘light’ molecules or small fragments of larger molecules such as acetate and formate. While low TOC means that the UPW system is working effectively to eliminate the source water organic load, this test is not an accurate measure of sterility of a UPW system. In addition, TOC levels at POU can also reflect carryover from chemical baths and contaminants in cleanroom air.

8.3 *Dissolved Oxygen (ppb)*

8.3.1 Unless dissolved oxygen (DO) content is controlled, rinsing hydrogen-passivated silicon wafer surfaces with high DO UPW can result in etching of the silicon by the oxygenated UPW and loss of control of gate oxide thickness. Dissolved oxygen is measured online by two basic technologies: electrochemical cell or optical fluorescence. Traditional electrochemical measurement uses a sensor with a gas-permeable membrane. Behind the membrane, electrodes immersed in an electrolyte develop an electric current directly proportional to the oxygen partial pressure of the sample. The signal is temperature compensated for the oxygen solubility in water, the electrochemical cell output and the diffusion rate of oxygen through the membrane. Optical fluorescent DO sensors use a light source, a fluorophore and an optical detector. The fluorophore is immersed in the sample. Light is directed at the fluorophore which absorbs energy and then re-emits light at a longer wavelength. The duration and intensity of the re-emitted light is related to the dissolved oxygen partial pressure by the Stern-Volmer relationship. The signal is temperature

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compensated for the solubility of oxygen in water and the fluorophore characteristics to obtain the DO concentration value. Refer to ASTM D5462 for DO testing.

8.4 *Particulate Matter (Particles/L)*

8.4.1 Particles that adhere to wafer surfaces at each step of the integrated circuit device manufacture may impair the application of thin-films and photolithographic substances and ultimately cause discrete and integrated transistors to fail because of resultant physical imperfections

8.4.2 *Sources of Particles in Ultrapure Water Supplies*

8.4.2.1 Particles originate in the incoming city water and may also be released from UPW system materials. Incoming source water contains a high level of particles, which are removed by prefilters and mixed media in order to remove gross physical turbidity in the micron range.

8.4.2.2 After RO source water particles are greatly reduced because the RO membranes also reject particles. It is essential to minimize the particle load to the RO to prevent membrane fouling and premature or frequent RO cleanings. From the RO outlet forward (in the system) the particle load in the UPW originates from system components. Particle sources can be RO membrane modules, piping components, valves and other similar control devices, tank linings, resin fines and other sources where materials are undergoing wear or degradation.

8.4.2.3 After the ion-exchange resin beds, filters of increasingly smaller dimension are used in the range 0.2 to 1.0 microns to remove residual particles and resin fines. Most ultra-high purity applications use ultra filtration at 10,000 molecular weight, or smaller. In most UPW systems, the presence of significant quantities of sub-micron particles at the final filter may be due to shedding of upstream UPW system treatment systems' materials. In addition, POU filters on tools can be a source of contamination if not maintained.

8.4.3 *Particle Monitoring*

8.4.3.1 SEMI F63 provides detailed discussion of the available particle monitoring methods and their ability to support the advanced semiconductor industry technology needs. ASTM D5127 refers to laser optical particle counting (OPC) and scanning electron microscopy (SEM).

8.4.3.2 OPC technology have traditionally been used for trend analysis and specification compliance. However, advances in OPC technology have not kept pace with the ever narrowing line-widths of the latest semiconductor manufacturing technology. OPCs can monitor particles in the 0.02 to >0.5 micron range but the detection efficiency at the rated lowest size is only a few percent at best. In order for an OPC to 'see' a particle there has to be a significant difference between the refractive index of the particle and the refractive index of water. Therefore a bacterium, composed of about 90% water, is invisible to an OPC. However, even with these limitations OPC can still play an important role in monitoring the UPW quality by monitoring the performance of less-demanding filters. Particle counting using an OPC requires a good sample port connection to eliminate false counts. Good counter maintenance, such as performing routine calibration and replacement of tubing and fittings is critical.

8.4.4 *SEM Analysis or Direct Count Analysis of Particles and Bacteria*

8.4.4.1 SEM is used to analyze particles and bacteria within the UPW system. Three major features of the SEM method are the enumeration of particles, enumeration of bacteria and the determination of the elemental composition of the captured particles. Depending on the particulate level in the UPW, relatively large volume of UPW is filtered online, typically at the POD. Due to particles created by the act of sampling and the pre-existing particles found normally on a new filter prior to use, a volume of over 1000 liters may be required to get a statistically valid result. In the laboratory, the filter is removed from the filter holder and placed in the vacuum chamber of an SEM instrument. A focused beam of electrons is scanned across the filter, systematically allowing the size, shape and distribution of the particles to be measured. The electron microscope is capable of extremely high power detection (10,000× magnification), and the counting process may be automated. Bacteria and particles in the >0.05 micron range are detected, counted, and reported in units of particles per litter taking into account the filtered sample volume.

8.4.4.2 When SEM particle capture filters are installed at critical areas in the UPW system on a continuous basis, they may be used to determine the source and nature of intermittent particle excursions. Combination of monitoring by OPC and SEM is becoming particularly important in light of limited capability of the existing methods to control particles at size defined for critical particles (smaller than metrology capability).

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8.4.4.3 New nonoptical online particle counting methods are currently under development with the potential of monitoring particles smaller than 10 nm.

8.4.5 *Identification of Particles*

8.4.5.1 As part of the SEM analysis, an energy dispersive x-ray beam (EDS) may be applied to a particle to produce a spectral output of the composition of the particle. EDX results allow the basic classification of organic and inorganic components. Elemental compositions are provided that can be used to trace the source of particulates. For example, a particle having Cr and Fe peaks is probably from a stainless steel source while one having C and F peaks is probably composed of fluoropolymer materials. The ability to use EDX for particle element composition is limited to certain size of the particles, depending on the capability of the SEM-EDS used. Below this size the amount of X-ray energy dispersed becomes too small to detect.

8.5 *Bacteria*

8.5.1 *Sources of Bacteria in Water Supplies*

8.5.1.1 Incoming city water supplies and UPW system components are all potential sources of biological contaminants. Viable (live) counts are usually low in city water due to chlorination. Chlorine removal, chemical addition, material leach outs, and low flow areas in the components are contributors enabling biological growth within an UPW system. Fouling of RO membranes and ion-exchange resins may occur, requiring costly remediation.

8.5.1.2 The oligotrophic nature of UPW causes bacteria to become hydrophobic which creates a thermodynamic adhesion to the wall. Nutrients are brought to the adhered bacteria by the water flowing past them and they multiply, creating a biofilm. Planktonic bacteria remain in the water flow and are less of a challenge. While some system owners employ sanitization programs in hopes of safeguarding against microbial activity, biofilm can prove resistant and may permanently coat the inaccessible surfaces of valves and dead-legs. Despite the fact that UPW systems are designed to be hostile to most bacterial species, the formation of biofilm on filters, in membranes, and in ion-exchange resins is widespread and periodic flourishing of bacterial colonies can be a costly long-term problem.

8.5.2 *Viable Bacteria by Culture*

8.5.2.1 Replicate samples are collected in sterile containers at each sampling point, passed through special sterile filters, and then dosed with a growth medium. In an incubator, bacteria are cultured and grow to form colonies. The bacteria colonies are counted under low power magnification after a specified interval and at a specified temperature. Refer to ASTM F1094 for bacteria testing method. Results may vary depending upon factors such as sample volume, growth media, incubation time and temperature, and enumeration methods. It may be necessary to evaluate different methods to determine the best recovery for a specific UPW system. Bacteria results are reported as colony-forming units (CFU) per unit volume (e.g., 100 mL or 1000 mL). For very low bacteria UPW systems, larger sized samples may be collected to provide lower detection limits. Larger volumes can be filtered using this method, making the bacterial counts more statistically significant.

8.5.3 *Total Bacteria by Epifluorescence*

8.5.3.1 After collection, samples are filtered onto polycarbonate membranes, which are then stained with dyes that cause biological materials to fluoresce under ultraviolet light. Using a high-power microscope live and dead bacteria are identified and a counting method is used to give statistical accuracy for the sample size. This test provides accurate information about bacterial content (both viable and non-viable) of the UPW system rather than an estimate of bacteria that is capable of being cultured during a growth opportunity (the culture method). On-line samples may also be obtained using a method similar to that of the SEM particle capture method. Larger volumes can be filtered using this method making the bacterial counts more statistically significant. The culture method may also be performed concurrently to determine if an increase in levels is due to live or dead bacteria.

8.5.4 *Total Viable Organisms by Scan RDI*

8.5.4.1 Scan RDI is a semi-automated method of measuring total viability counts. The method is able to detect a single cell based on direct measurements of cell activity and includes bacteria and other live organisms that may be present in biofilm.

8.5.4.2 The sample is filtered through a membrane and a counterstain is added to minimize background fluorescence. Viable organisms are labeled using a nonfluorescent substrate that diffuses across the cell membrane. The labeling differentiates between viable and dead cells based on the presence or absence of esterase activity and intact cell

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membranes. Only viable cells with membrane activity have the ability to perform this cleavage and retain the fluorescent label. No cell growth is required. The membrane is then scanned by a solid-phase laser-scanning cytometer. The cells are detected, counted and verified within minutes.

8.6 *Silica*

8.6.1 *Forms of Silica in UPW Systems*

8.6.1.1 Silica exists primarily in two forms: as silicates and as polymeric silica. Silicates are referred to as dissolved silica and are reactive to molybdate using the heteropoly method (see ASTM D859). The polymeric forms are called colloidal silica or particulate silica and are not measured directly by the heteropoly method. As pH in a UPW system is lowered, silicates polymerize to form colloidal silica, which range in size from a few nanometers to several tenths of a micron.

8.6.1.2 As with TOC, RO membranes are responsible for the gross rejection of silica in incoming waters. Within the UPW system, dissolved silica is further removed in the anionic ion exchange resin beads. If conditions in an ion-exchange bed are favorable, such as a low pH, dissolved silica can be polymerized to form colloidal silica. However, colloidal silica is only weakly attracted to the anionic resins and is one of the first species to be liberated when the resin beds approach exhaustion. After anion resins are exhausted, increasingly high levels of silica (both dissolved and colloidal) are released into the UPW stream. Since colloidal silica is only weakly charged, the on-line resistivity meter is not sensitive enough to detect colloidal silica leakage prior to total resin bed exhaustion (i.e., breakthrough). It is very important to detect this onset of silica breakthrough before a drop in resistivity.

8.6.2 *Dissolved Silica Techniques*

8.6.2.1 A colorimetric heteropoly blue method is used to measure dissolved silica. Dissolved silica reacts with the molybdate reagent at acidic pH and forms a yellow complex in direct proportion to concentration. A reducing agent is added to the prepared sample, causing a blue color to develop. This color change can be quantified by a sensitive spectrophotometer for ppb to sub-ppb levels of dissolved silica. While a grab sample is often used as cost effective and allowing for reaching lower detection limits, on-line instruments may provide continuous monitoring of UPW systems. Ion Chromatography may also be used to measure reactive silica at relatively low detection limits. Ion chromatography in this case is used as a way to concentrate silica with subsequent detection using heteropoly blue method.

8.6.3 *Total Silica Technique*

8.6.3.1 Since all forms of silica are not reactive in the heteropoly method, the quantity of total silica in UPW must be verified independently. Total Silica may be measured by graphite furnace atomic absorption spectroscopy (GFAAS), inductively coupled plasma atomic-emission spectroscopy (ICP-AES) or inductively coupled plasma-mass spectrometry (ICP-MS) at ppb levels. Techniques such as ICP-MS and GFAA are capable of quantifying the silicon present to sub-ppb levels after pre-concentration. The method detection limit of total silica obtained by this technique is in the range of 0.5 to 1 ppb. Colloidal silica may also be chemically digested, converting it into reactive silica with subsequent heteropoly blue method analysis.

8.6.4 *Colloidal Silica*

8.6.4.1 The difference between total silica and dissolved silica may be represented by the colloidal form of silica. Please note, when colloidal silica analysis is required, it is important to take into account the fact that near the limit of detection of fundamentally different methods of reactive and total silica analyses, there may be increasing deviation often resulting in either negative values or unrealistically high ones.

8.7 *Nonvolatile Residue (NVR)*

8.7.1 Nonvolatile residue in UPW primarily consists of dissolved inorganic material. In the final polished UPW the most likely dissolved inorganic material is silica in both the dissolved and colloidal form. The primary source of this breakthrough silica is the anion resin bed as it approaches exhaustion. Sub-ppb levels of dissolved silica can be detected as non-volatile residue. There is also evidence that as an ion-exchange bed approaches exhaustion, previously removed dissolved silica can be subsequently released as colloidal silica if the pH in the ion-exchange bed is favorable. There is currently no specific measurement of on-line colloidal silica but detecting it as a component of nonvolatile residue can be beneficial.

8.7.2 *Method of Measurement for NVR*

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8.7.2.1 Refer to ASTM D5544 for NVR method. The online measurement of nonvolatile residue is based on the principle of determining 'residue after evaporation' of atomized ultrapure water droplets. Droplets evaporate in a fraction of a second leaving an ultrafine nonvolatile residue particle agglomerate a few nanometers in diameter. The size of the particle is related to the amount of residue impurity originally present in the ultrapure water. A condensation particle counter then monitors the concentration of ultrafine nonvolatile residue particles. The measurement range of this technique is 0.01 parts per billion to 20 parts per million.

8.8 *Ions*

8.8.1 *Mass Balance and Ionic Removal in UPW Systems*

8.8.1.1 The ionic composition of natural waters is in a dynamic equilibrium (mass balance) with the geological source. The total dissolved solids (TDS) content is composed of negatively charged (anionic) and positively charged (cationic) species that can be analyzed by ion chromatography (IC). RO membranes, ion-exchange resins, and electronic deionization (EDI) are used to remove ionic species from water. The UPW resin beds must be monitored because each unit has a fixed capacity to remove ions over time. Weakly bound singly charged (monovalent) ions such as Na^+ and Cl^- are continually displaced in favor of more highly charged doubly charged (divalent) ions such as SO_4^{2-} and Ca^{2+} throughout the life of the resin. When ionic breakthrough occurs the weakly bound ions are liberated in excess and resin beds must be regenerated.

8.8.2 *IC Techniques*

8.8.2.1 Ionic (anions and cations) removal in UPW systems can be quantified at very sensitive levels using the technique of ion chromatography (see ASTM D4327). During IC analysis a UPW sample is injected into an analytical column packed with a special ion-exchange resin. The ions from the samples are eluted (washed) down the column by a suitable eluent. Depending on their binding energy and sizes, the ions move down the column at different speeds. In time, all of the ions are separated and measured by a conductivity detector. External calibration of a sensitive conductivity or other detectors across a wide range of concentrations allows accurate quantification of anion and cations. Typical anions and cations determined by IC are fluoride (F^-), chloride (Cl), nitrite (NO_2^-), bromide (Br^-), nitrate (NO_3^-), sulfate (SO_4^{2-}). phosphate (PO_4^{3-}) and lithium (Li^+), sodium (Na^+), ammonium (NH_4^+), potassium (K^+), magnesium (Mg^{2+}), calcium (Ca^{2+}) respectively.

8.8.3 *Ions Critical in Semiconductor Processing*

8.8.3.1 Some types of haze formation on wafer surfaces may be caused by high concentrations of anions and cations on the silicon surface as residue. However, there could be other causes for haze formations therefore testing of anions and cations at the POU in the rinse bath may be important to identify and prevent this source of contamination.

8.9 *Metals*

8.9.1 *Measurement Methods*

8.9.1.1 Up to 67 metals may be determined by instruments such as GFAAS, ICP-AES or ICP-MS.

8.9.2 *Sources of Trace Metals*

8.9.2.1 Trace metal concentrations in feed waters vary over orders of magnitude according to the geology of source rocks and the residence time of water in aquifers and surface systems. Surface water supplies are often mixed by the local municipal services and seasonal variations are also introduced as ground water and well waters are brought in and out of service. Consequently, periodic analysis of metals in incoming feedwater and at key points of the UPW system (including after the FF) is essential so that significant changes in performance may be corrected early in the process of deionization.

8.9.2.2 Even though trace amounts of metals may be well controlled by UPW treatment system, there is still a risk of metallic contamination coming from materials of construction used in the distribution system. It is recommended that SEMI F57 is effectively used for the quality of high purity polymer material qualification.

8.9.3 *Boron*

8.9.3.1 Boron is a trace element that, like silica, is weakly attracted to anion resins. Both elements serve as a flag for the onset of ionic breakthrough through the anion exchange resin. A rapid and systematic rise in the boron content immediately after the resin bed is typical when ion-exchange resins approach exhaustion in the UPW system. Silica

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and boron breakthrough rates and each element's selectivity in the ion exchange process is a matter of ongoing study. The safest route is to measure both, but at least to focus on the one most prevalent in the city water feed stream.

8.9.4 *Contaminants from the Distribution System*

8.9.4.1 Certain heavy elements such as Zn, Mn, Ni, Cu, Cr, and Pb may be present as trace impurities in plastic (PFA and PVDF) components and may continuously leach into the UPW streams. Plastic-lined metal components such as pumps, storage tanks, and other UPW processing vessels may develop pinholes, thereby leaching out heavy metal impurities into the UPW system.

8.9.5 *Contaminants from Feed Water*

8.9.5.1 The alkali metals (Na, K), alkali earths (Mg, Ca) and Fe comprise the bulk elements of most natural waters and play a significant role in bulk water chemistry including hardness, alkalinity and total dissolved solids. Of these elements, sodium is the most abundant and is present in final filter UPW at sub-ppb level. Highly charged metals (Al, Fe, and Mn) have the potential to coagulate and foul RO membranes, and Ba and Sr can cause scaling of the RO and ion exchange resins.

8.9.6 *Radioactive Elements*

8.9.6.1 Radioactive elements such as uranium and thorium can cause soft X-ray damage to devices when present at ppt levels. These elements are detectable in a full ICP-MS scan.

9 Typical Monitoring Programs

9.1 *Online Monitoring*

9.1.1 Online monitors for UPW are readily available from a variety of manufacturers. Before selecting an instrument for online monitoring an evaluation should be made about (1) sensitivity of measurements, (2) accuracy of the measurements, (3) reproducibility of the measurements, (4) calibration protocols for the instrument, (5) noise level or zero count for the instrument (particle counters) and (6) instrument cost and reliability.

9.1.2 If online monitors are not available at critical sampling points or the sensitivity of the online monitor is not sufficient to accurately measure at the required levels, grab samples may be taken and sent to a qualified laboratory for equivalent testing. Use of ISO 17025 certified laboratory is recommended. This offline sampling and testing requires special apparatus and trained personnel. A clear understanding of the limits of each method is required before its application.

9.1.3 All online monitoring requires clean and tight sample port connections in order to eliminate false counts.

9.2 Testing at critical sampling points, such as POU, requires special sampling apparatus and trained personnel. The enclosures and mini-environments around wet benches that are designed to prevent accidental access and to isolate human contamination from the wafer can make it difficult to sample the bath. However, contamination-free sampling can be done in a safe way using high purity fluoropolymer apparatus and trained qualified personnel.

9.3 The utilization of unbiased, independent and experienced personnel in obtaining data at critical sampling sites with the UPW system where on-line instrumentation is not available is essential in determining the overall performance of the UPW system.

9.4 See Table 1 §9 UPW System Test Parameters, Sampling Points, and Recommended Monitoring Frequencies for sample point and monitoring frequency recommendations.

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Table 1 UPW System Test Parameters, Sampling Points and Recommended Monitoring Frequency for Assuring Good UPW System Operation

	Incoming Feed pre RO filters	*Pre RO* *Post RO prefilters*	*Permeate* *Post RO*	*Make-up* *Post Ion Exchange*	*UPW POD* *Post Final Filter*	*UPW* *Return Loop*	*UPW POC or POU (Tools) (Recommended)*
Resistivity	-	Continuous	Continuous	Continuous	Continuous	Continuous	-
TOC	Monthly	Bi-Weekly	Continuous	Continuous	Continuous	Continuous	Bi-Weekly
Particles OPC	Online (turbidity)	Trouble-shooting only	Trouble-shooting only	Trouble-shooting only	Continuous	Continuous	Continuous
Particle SEM	-	-	-	Trouble-shooting only	Monthly	Monthly	Monthly
Bacteria	-	-	Trouble-shooting only	Trouble-shooting only	Weekly	Weekly	Weekly
Dissolved Silica	Monthly	-	Monthly	Continuous	Continuous	Monthly	Monthly
Total Silica	Monthly	-	Monthly	Weekly	Weekly	Weekly	Trouble-shooting only
NVR	-	-	-	-	Continuous	Trouble-shooting only	-
Sodium	-	-	-	Continuous	Monthly	Monthly	Trouble-shooting only
Boron	Monthly	-	-	Monthly	Weekly	-	-
Trace Metals-Full Scan	Monthly	-	-	Monthly	Monthly	Monthly	Trouble-shooting only

#1 The frequency noted for these parameters is not intended to assure against excursions due to improper maintenance procedures or other external means of system contamination

10 Choice of Instrumentation

10.1 Make sure the selected instrument is appropriate for its intended use and application. Before specification of the analyzers, it is important to know the targeted UPW system concentration specifications for the analytical instruments, DO, Ozone, TOC, Particle Counters, Sodium Analyzer, Conductivity, Peroxide, NVR and others. SEMI F63 provides a list of recommended UPW quality parameters.

10.2 It is important that the limits of quantitation for the instruments are good enough to measure at the targeted concentration specification levels. Consult the instrumentation suppliers for the limits of quantification.

10.3 Each UPW system process step needs to be reviewed for its expected (target) performance. Key performance parameters should be defined and the need of the instrumentation to monitor those parameters should be evaluated. Table 1, §9 and Table 2, §13 provide examples of the typical performance parameters at each process step and their respective monitoring instrumentation recommendations.

10.4 Critical final UPW quality instruments should be defined by the semiconductor company UPW experts based on internal specifications (if these exist) or on SEMI F63 recommendations.

11 Recommended Analytical Instrument Placement Locations in a UPW System

11.1 See § 13, Table 2 and Figure 1 for recommended analytical instruments and sampling locations.

NOTE 6: Table 2 is offered as basis for specific discussion and determination for what type and at what locations that metrology should be used.

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12 Installation and Validation

12.1 Follow analyzer or sensor vendor's installation and validation instructions and procedures.

12.2 Select appropriate sampling connections and sample tubing.

12.3 Select appropriate combined sampling system and analyzer UPW rinse times to reach operational specifications.

12.4 Design sampling manifold to prevent sampling interference from other analyzers or to cause interference in other analyzers or sensors.

13 Operational

Ensure that the following is in place:

13.1 Scheduled Maintenance per vendor recommendation and per data output for each analyzer or sensor.

13.2 Scheduled Calibration verifications per sensor or analyzer vendor recommendations and per data output.

13.3 Consider providing backup sensors or analyzers based on the following:

13.3.1 Planned Instrumentation maintenance calibration downtime.

13.3.2 Unplanned Instrumentation downtime.

13.3.3 Redundant and/or portable sensors/analyzers for process debugging and water system component replacement/maintenance rinse up/rinse down control.

Table 2 Recommended Analytical Instruments and Sampling Locations for a Semiconductor UPW System

Analytical Instrumentation List	#	Locations
Pretreatment		
Chlorine Analyzer (optional)	2	Before and After Activated Carbon or Chlorine Removal
TOC Analyzer (suitable for non-deionized water)	1	Incoming feed water source
Conductivity sensor	3	Incoming feed water source and RO permeate and reject headers
Hardness Analyzer (important only for those applications where even traces of hardness are detrimental)	2	Incoming feed water source, and post softening
Alkalinity Analyzer (optional)	1	Incoming feed water source
Silt Density Index (Portable) Analyzer	1	Portable for profiling each filtration step
pH Sensor	2	Incoming feed water source, and RO inlet header; dual sensors may be considered for critical applications where active pH control is used.
ORP Sensor	1	RO inlet header
Ultrasonic Precision Flow Sensors	4	Incoming feed water, RO inlet, RO outlet, and RO Permeate headers
Temperature Sensor	1	RO inlet header (dual sensors may be considered for critical applications where active temperature control is used).
Pressure Sensor	1	RO Inlet header
Primary		
Online Boron Analyzer (with sequencer)	1	Primary Anion bed or Mixed Bed outlet header
Sodium Analyzer (with sequencer)	1	Primary Mixed Bed outlet header
TOC Analyzer (suitable for deionized water)	1	Primary Cartridge Filter Outlet Header
Ultrasonic Precision Flow Sensors	3	Primary Supply (to UPW), Return and Rinse Headers
Resistivity Sensors	5	Makeup feed to Primary Storage Tank, Primary TOC UV Outlet Header, Primary MB Inlet and Outlet headers, Primary Supply (to UPW)

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Analytical Instrumentation List	#	Locations
Temperature Sensor	2	Primary Supply and Return
Pressure Sensor	2	Primary Supply and Return
Polish Cold		
TOC Analyzer (suitable for deionized water)	1	Point of Distribution
On-line non-volatile residue monitor	1	Point of Distribution
On-line Particle Counter	1	Point of Distribution
Dissolved O_2 Analyzer	1	Point of Distribution
Dissolved N_2 Analyzer	1	Point of Distribution
Ultrasonic Precision Flow Sensors	TBD	(number is system specific) – Typical: Distribution Pump Outlet Header, Point of Supply, Point of Supply Shunt Valve, End of Supply Distribution Main Header, End of Return Collection Header
Resistivity Sensors	TBD	Point of Distribution (number is system specific)
Temperature Sensors	2	Point of Distribution
Pressure Sensors	TBD	Point of Distribution (number is system specific)
Hot UPW		
TOC Analyzer (suitable for deionized water)	1	Point of Distribution
On-line non-volatile residue monitor	1	Point of Distribution
Particle Counter 20 nm or smaller	1	Point of Distribution
Ultrasonic Precision Flow Sensors	TBD	(number is system specific) – Typical: Distribution Pump Outlet Header, Point of Supply, Point of Supply Shunt Valve, End of Supply Distribution Main Header, End of Return Collection Header
Resistivity Sensors	3	HUPW HX Outlet header, Point of Supply and End of UPW Return Collection Header
Temperature Sensors	5	Recovery HX Inlet and Outlet Headers, HUPW HX Outlet Header, HUPW Point of Supply, End of Supply Distribution Main Header
Pressure Rosemount Sensors	TBD	(number is system specific) – Typical: Between all unit operations, Point of Supply, End of Supply Distribution Main Header(s), End of Return Collection Header(s)

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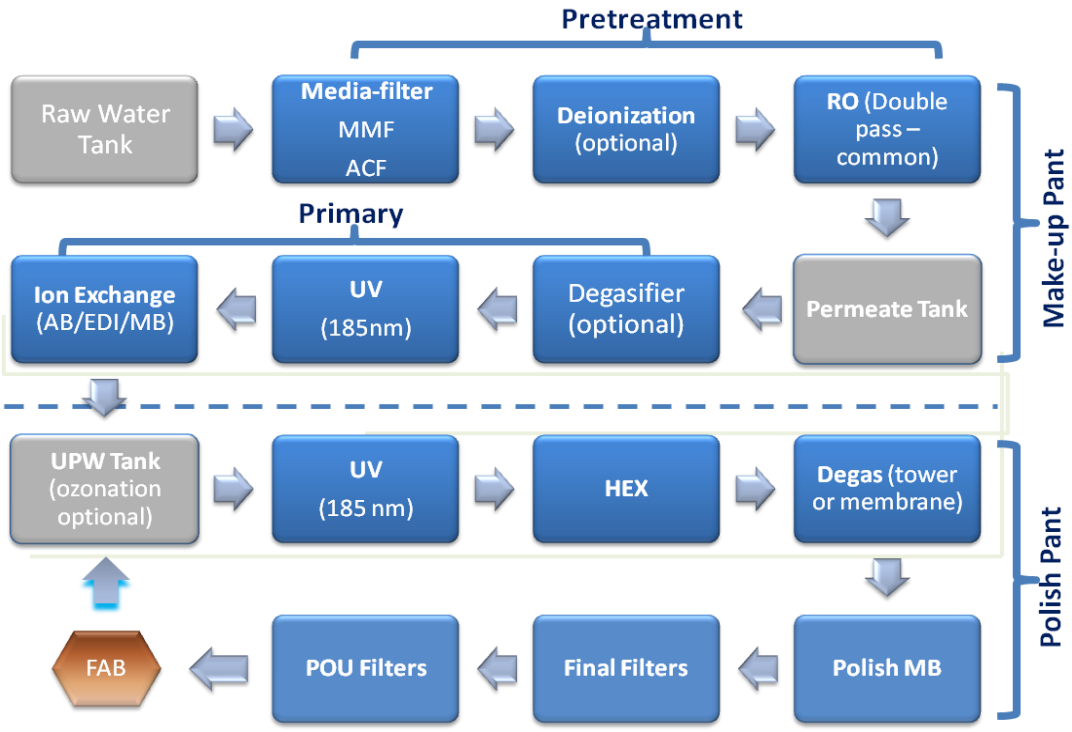


Figure 1
A Typical Semiconductor UPW Configuration

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