SEMICONDUCTORS

DETERMINING SEMICONDUCTOR WATER QUALITY GUIDELINES (TODAY AND TOMORROW)

High-purity water guidelines for semiconductors are a tool for facilities managers and process engineers to ensure that the supply is consistent and does not negatively impact the final product. This article examines key factors that should be understood and taken into account in setting these guidelines.

As geometries shrink to 0.09 micron (μm) and beyond, water quality discussions by active organizations today such as American Society for Testing and Materials (ASTM), Semiconductor Equipment and Materials International (SEMI), International Technology Roadmap for Semiconductors (ITRS), and Air Liquide - Balazs Analytical Services (Balazs) continue to help identify process needs. From the beginning, high-purity water guidelines have focused on water quality at the facilities point of distribution (POD). New sampling locations are now emerging to better define water quality needs closer to the wafer point of use (POU).

Because we rely so heavily on analytical results when setting specifications and in making operational decisions, it is critical to understand what these numbers represent. Some current and future water quality guidelines are driving detection limits closer to edge of the analytical tools' capabilities. To ensure that the user of these guidelines understands what the numbers represent and to help make the best operational deci-

sions, an overview of analytical methods and detection limit definitions is also presented.

Overview of Guidelines
For this article, specifications are defined as water quality requirements. They are or should be process specific. Guidelines are defined as a starting place for discussion and are meant to be modified, where appropriate, by the enduser.

Historically, high-purity water guidelines and specifications have been established using a number of different protocols. This includes back calculation from levels effecting wafer yield, as in ITRS. They have also been established by facilities with SPC to show that high-purity water production is under control, such as in SEMI F63. And they have been established by analytical detection limits, where any analyte that is detectable is deemed to exceed acceptable limits, and conversely, any analyte that is not detectable is deemed to be under control.

Currently, ITRS, SEMI, ASTM, and Balazs have generated public domain guidelines and specifications for electronic grade high-purity water, and corporations and facilities have established numerous in house limits. By attempting to bring all of these considerations together, we'll be in a better position to work toward establishing current or future guidelines.

Sampling Locations Review
Water quality guidelines for the microelectronics industry have historically aimed towards best achievable and best-measurable levels of water quality

at the point of distribution. With stable, high-quality water in place at the POD, the industry is shifting its focus to study water quality closer to the wafer POU.

Continued monitoring of the POD location assures the customer that the water plant is operating correctly and high-purity water is consistently discharged. In addition to the POD, many supporting locations are also monitored within the purification system to ensure quality compliance. Monitoring the incoming water, major water system components, and the return water to the plant is important to detect quality changes before they negatively impact the POD. In addition, PODs are relatively easy to sample and a large database of information is available.

Recently, new locations have been proposed for applying high-purity water guidelines. The 2004 ITRS guidelines have moved closer to the wafer POU by proposing limits at the tool point of connect (POC). As high-purity water travels through the distribution system and components to the tool, the water quality may be compromised before reaching the tool POC. On a well-balanced, well-plumbed distribution system, the degradation will be minimal. However, little baseline data has been collected to study water quality fluctuations at the POC due to sampling ports not being available or easily accessible, and the large number of potential sampling sites in a fab. Since even more sampling challenges exist at the POU, the POC offers a feasible alternative with easier access for sampling. At the POC steady water pressure is available, allowing existing on-line and off-line sampling techniques to be used.

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TABLE A
Factors to Consider in Analytical Method Selection

Required	*Desirable*
Method Bias	Speed
Precision	Low Cost
Accuracy	Ruggedness
Sensitivity	Ease of Operation
Selectivity	

SEMI has developed a guideline requiring rinsing and testing for all assembly units installed in process equipment before shipment of these units to tool manufacturers or to the fab (SEMI E49.2). This procedure tests potential impacts to water quality between the POC and POU. Challenges for SEMI E49.2 include possible redesign of assemblies to allow samples to be pulled, the potential need for specialized sampling techniques, and in some cases, multiple sampling points within a tool.

As geometries shrink below 0.09 µm, discussions will continue to determine the best ways to meet wafer-processing needs. Although the POD will remain an important monitoring location to guarantee optimum high-purity water system performance, analyzing parameters at lower detection limits will become more critical to detect water quality changes before impacting product yields.

With stable POD water quality the norm, it has become important to shift the focus of future guidelines from the POD to the wafer POU. Understanding water quality fluctuations originating from semiconductor cleanroom environment, distribution lines, tool hardware, design and operation is the precursor to correlating data to process yields. In addition, with high-purity water production costs high and small improvements requiring large expenditures, facilities operational savings can also be realized with increased testing at the POU.

As previously noted, data collection at the POU has been limited for several reasons. Like the POC, there are many locations within a fab and each tool is different in design and process operation, offering many ways to impact final water quality. Unlike POC, a steady water pressure and easy access to distribution lines are not available for sampling at the wafer. New sample collection methods will need to be developed before water quality within a tool can be accurately analyzed on a routine basis. As we move toward the future, an industry task force consisting of process engineers, facilities, analytical lab, and tool manufacturer personnel will be essential to effectively address and solve the many POU sampling challenges.

An Inside Look
When a laboratory evaluates analytical methods, the lab must pick those that will generate reliable data with sufficient sensitivity to be useful to an enduser.

	MDL[1] (ppt)	Confidence Interval[2]	RSD[3] %	RL[4]	Confidence (ppt)	RSD[3] % Interval[2]
TABLE B Example of Statistical Summary						
Element						
Calcium	0.9	±0.46	43.3	2	±0.21	7.0
Iron	0.8	±0.34	22.4	2	±0.19	5.9
Nickel	0.8	±0.65	76.8	2	±0.45	13.7

Notes:
[1]Statistically determined method detection limit (parts per trillion).
[2] 95% confidence interval.
[3] Percent relative standard deviation of results obtained using several different low-level standards.
[4]Reporting limit verified by reading actual low-level standards (ppt).

When choosing a method the lab must strike the proper balance between required characteristics and desirable characteristics of the method. Table A summarizes the factors considered when selecting an analytical approach.

Generally speaking, there are two classes of test methods available to high-purity water analytical laboratories. These include routine or production methods, and research or non-routine methods. Each of these represents different approaches and philosophies towards monitoring, and together they give the operator and users of systems a more complete understanding as to how the system is performing. Coupled with additional information, including process data and product yields, this data can shed some insight as to whether or not a particular parameter is impacting the product.

Unless high-purity water is to be analyzed in a pure research mode, all routine analytical methods are optimized to take into consideration each of the required characteristics while being mindful of costs and turn-around-time (TAT) requirements. This typically results in some compromise to the reporting limit (RL) enabling the laboratory to process large numbers of samples in a timely and cost effective manner.

On the other hand, increases in sensitivity and/or more of a detailed characterization of a sample's constituents will generally call for alternate methods that require additional time, dedicated equipment, and a higher cost.

In understanding analytical data, it is important to know how the data will be used, the confidence in the numbers (± error bars), how detection limits are determined, and how all of the above should affect specifications.

Analytical Methods as a Tool
A properly balanced monitoring pro-

gram will take advantage of the best of both of these approaches. Using what are now considered fairly routine methods, a competent trace laboratory can quickly produce accurate data at the low-parts-per-trillion (ppt) level on a significant number of samples. The value of this testing is that it provides a sufficient number of data points that serve as indicators that the system is operating properly. As part of a regular testing program, this data also assists facilities personnel in detecting changes in the plant's performance early on. This data ties directly into the guidelines, which are geared towards the operational side of the process. This sort of information allows the operator to make adjustments quickly and efficiently.

However, many of these routine methods will not provide the sensitivity required to meet the new ITRS specifications, which are pushing single-ppt levels of quantitation. For some parameters, less routine methods will be required. In many cases, these methods already exist, but as previously noted above they require an extended TAT and a higher cost, which translate into fewer samples. To get the most benefit from this sort of testing, samples need to be carefully chosen to ensure that the information obtained is useful when trying to tie the water quality to the actual product produced.

What Does Your Result Mean?
Pushing analytical data to the extreme levels of sensitivity required to hit some ITRS specifications puts a burden on the lab to ensure that they are producing both accurate and precise data. Additionally, the lab must understand and be able to communicate the uncertainty associated with the data reported. Only then can the user of the data act with confidence in making decisions related to their system or process.

Simply getting a lower detection limit is only a small part of the process involved in evaluating a system. It is incumbent upon the user of the data to fully understand how detection limits have been determined and what the confidence limits are.

Additionally, the laboratory's reliability checks and routine quality control (QC) procedures must be clearly stated. Only by understanding how a lab defines its data and monitors itself will the data user be in a position to make effective go/no go decisions. The usefulness of the data is obviously enhanced if the uncertainty of the data is understood.

For example, the instrument detection limit (IDL) is a measure of the performance of the analytical tool under optimum conditions with no impact from sample handling or matrix effect. By contrast, the method detection limit (MDL) incorporates sample container, prep, and matrix issues (just as would be the case with a sample) into the determination. The IDL is always less than the MDL. Unfortunately, because both IDLs and MDLs are statistically based, they may not be achievable on a daily basis. The signal-to-noise ratio may not even be sufficient for detection. Statistics are set to limit chances of a false positive, not a false negative. Generally, neither one takes impacts from sample collection into account.

Data reported at the IDL or MDL look impressive, but the uncertainty of the data may be quite high (>200%). This data is not reliable for making operational decisions.

A practical approach is for the analytical laboratory to set a reporting limit (RL) at 2 to 5 times the MDL. The ability to actually see the analyte at the RL can then be verified by running a check standard at a concentration level near the RL. This means that the reported result would have a much greater degree of certainty associated with it and the user can have more confidence that the number has significance. However, although it is true that results at the RL are more accurate than results at the IDL or MDL, it is still possible for some results at the RL to still have an uncertainty of 50% or more. The bottom line is that even though there is some compromise to the lower level of reporting using an RL as opposed to a MDL, the user of the data can act with greater confidence and assurance that the results are actually of value.

Table B contains data contrasting the confidence intervals of statistically derived MDLs with RLs. The main difference between the MDL and the RL is that the RL was reproducibly measured at the analytical tool using actual low-level spikes whereas the MDL is simply a calculated value.

The data illustrates that there is a significant degree of uncertainty associated with each of the MDL values. In practice the uncertainty associated with the results reported at the MDL can easily be 100% to 200% or more. Although the confidence of values at the RL is tighter than at the MDL, the data also demonstrates that the degree of uncertainty varies from element to element. This factor must be considered when using the data and in setting guidelines.

Inevitably, the likelihood of a water system "failing" will increase if specs continue to be set at the MDL of an analytical method. It is recommended to set a spec limit of 10 times the RL for the parameter when possible. Unless one has confidence in and an understanding of the laboratory's practices, decisions should not be made on the basis of the data reported.

Setting a Guideline
As with laboratory "detection" limits, there are many ways that different organizations have come to develop guidelines. Table C is an example using calcium, which illustrates how different organizations at different times in recent history have addressed the issue of establishing guidelines.

Summary
In the past, water guidelines have focused on understanding water quality at the point of distribution while disregarding the wafer POU. A shift for our industry of looking at water quality at the tools has identified the need for method development of sampling methods to obtain useful samples at the POC and

POU. A consolidated effort by process and facilities engineers, analytical lab personnel, tool suppliers, and manufacturers will be necessary to establish guidelines at the wafer POU.

The industry continues to work towards a better understanding of water quality at the wafer POU and how it impacts yield. Several contaminant parameters have shown a negative impact on some wafer processes, and as a result, water quality guidelines are being adjusted based on this new information.

Incorporating a balanced analytical monitoring program, which uses both routine and state-of-the-art water testing methods will ensure facilities process control and safeguard product yields. To make the best decisions from this data, it is critical that the users of laboratory results fully understand how a lab generates its data and how that data is manipulated to arrive at the final reported number.

Finally, a reminder that guidelines should be used as a starting point for setting water quality specifications. Each site and process will need to develop a set of specifications based on their own specific internal requirements.■

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