Resistivity Instrumentation to Meet Today's Semiconductor Processing Requirements

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Abstract

Resistivity instrumentation has served the water purification industry well for many years. A few years ago, as a result of the reliability and endurance of this measurement, resistivity instrumentation was almost taken for granted when designing new water systems. It was not uncommon for resistivity instruments to be completely overlooked during the annual (or bi-annual) plant maintenance programs. Calibration was never seriously considered or, in some cases, instruments could not even be calibrated in the field. But today, with new technologies and improvements in water purification systems, resistivity instrumentation has become an integral part of a water purification system. Water purification designers are demanding greater reliability, lower costs, easier calibration processes, and improved accuracy.

Instrument manufacturers are now working on new resistivity measurement technologies to meet the needs of Ultrapure Water (UPW) systems well into the next century. Instruments are now available which incorporate new technologies that greatly improve and enhance instrument features. Today's instruments now have greater dynamic ranges that enable a single instrument to measure high resistivity fluids such as ultrapure water and highly conductive fluids such as acid and base concentrations. New circuit designs have improved the ability of the instrument to

measure temperature up to 10X better than older technology. Multiparameter instruments, originally introduced in the mid 80's, now have more input channels that allow even more measurements per instrument.

Introduction

Today's Microelectronics industry demands new and more accurate instruments with the ability to provide more measuring parameters and controls. The days when a resistivity instrument would provide a water system operator with only a simple analog resistivity indication are long gone. Modern resistivity instruments not only provide immediate digital resistivity indication with a higher level of accuracy, repeatability and measurement resolution, but they also have wider dynamic ranges and are much easier to operate and calibrate. Other process control features such as, analog outputs, relays, digital communication, and I/O lines make today's instruments much more effective. In fact, today's resistivity instrument manufacturers, driven by the microelectronic industry's need for cost reduction and their demand for additional analytical information, are producing instruments that can control complete sub-processes on their own.

Still, the primary requirement of a resistivity analyzer measuring UPW is the instrument's ability to detect ionic impurities at very low concentrations. Today's state-of-the-art water systems produce UPW with minimal ionic impurities. In fact, most modern systems produce UPW with ionic impurities below the detection limits of many resistivity instruments. This does not, however, render the resistivity instruments obsolete since they can detect catastrophic events and report them instantaneously.

Numerous technological advancements have recently been implemented to meet the demands of the microelectronics industry. The industry's need to detect lower concentrations of ionic impurities has driven instrumentation manufactures to invest more resources in research and development than previously. As a result, new instruments have been introduced that offer new and improved features. Some of the key features that newer instruments provide are:

- Better measurement accuracy
- More measurements per instrument
- More process control capabilities

A byproduct of the development investment has been additional features and enhancements such as:

- Traceable verification and calibration
- Easier installation
- User-friendly programming
- Versatile display options

These features make instruments more desirable and user-friendly to the operator.

This paper will focus on the instrumentation needs of the Semiconductor industry and the advances made by instrument manufacturers to meet those needs. It will address new technologies employed to improve accuracy and new feature and enhancements that facilitate operations.

Why is Accuracy Important

Semiconductor manufacturers must constantly strive for higher product yields and lower manufacturing costs if they are to remain competitive in today's market. Since water purity affects product yields, the need for reliable and accurate purity measurements is increasingly greater.

Resistivity, and its reciprocal conductivity[†], is one of the best methods to detect ionic contamination in a UPW water system. As a result its ability to repeatedly and accurately detect ultra-low levels of ionic contamination is critical to a semiconductor manufacturer. Ultrapure water has a conductivity of 0.0550 μS/cm at 25.00°C; therefore conductivity instruments must have the ability to accurately and repeatability detect very small conductivity changes on a non-zero background. Further complicating these measurement problems is the fact that the conductivity of the ions, formed from the dissociation of water, is dependent on temperature. Thus, if the water's conductivity increases, the measuring instrument must determine if the change is due to temperature increase, an increase in impurities, or a combination of both.

[†] Most of this section we will use the term "conductivity", since it increases with impurity concentration.

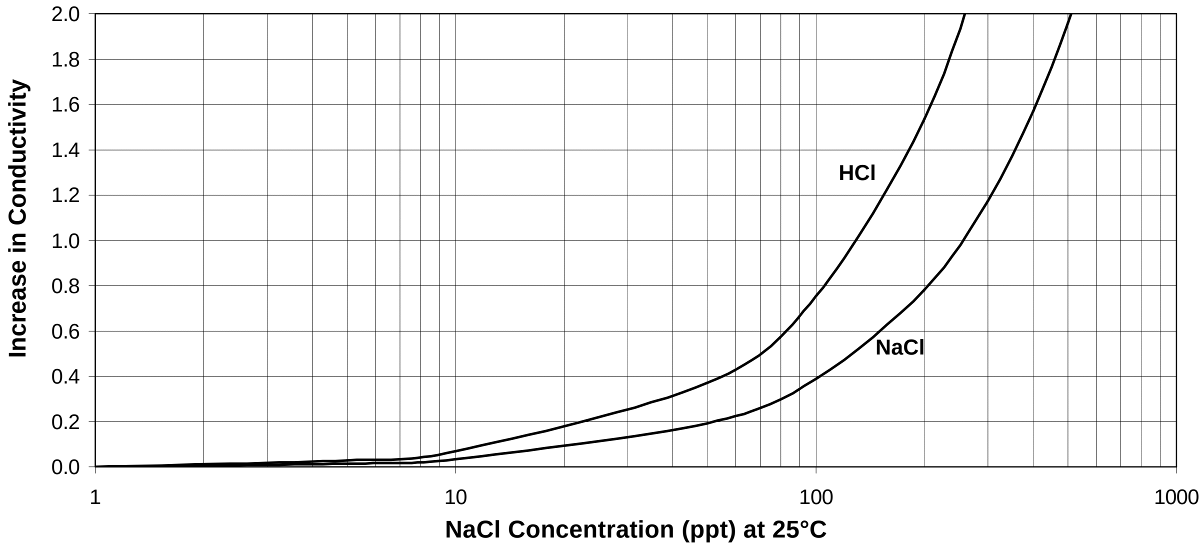
As demonstrated in Table 1, the problems are greatly magnified and more difficult to resolve at elevated temperatures. Although the conductivity change is greater at elevated temperatures for the same amount of impurities, the relative change is actually ~4X smaller. This makes far more difficult for a conductivity instrument to detect. The relative increase in conductivity as a function of concentration is shown in Figure 1.

Table 1. Conductivity of Ultrapure Water with Trace Impurities

[NaCl] (ppt)	25°C Conductivity Increase* (nS/cm)	25°C Conductivity Increase* (%)	80°C Conductivity Increase** (nS/cm)	80°C Conductivity Increase** (%)
1	0.002	0.004	0.005	0.001
10	0.022	0.039	0.051	0.011
100	0.217	0.391	0.506	0.108
1000	2.167	3.781	5.060	1.069

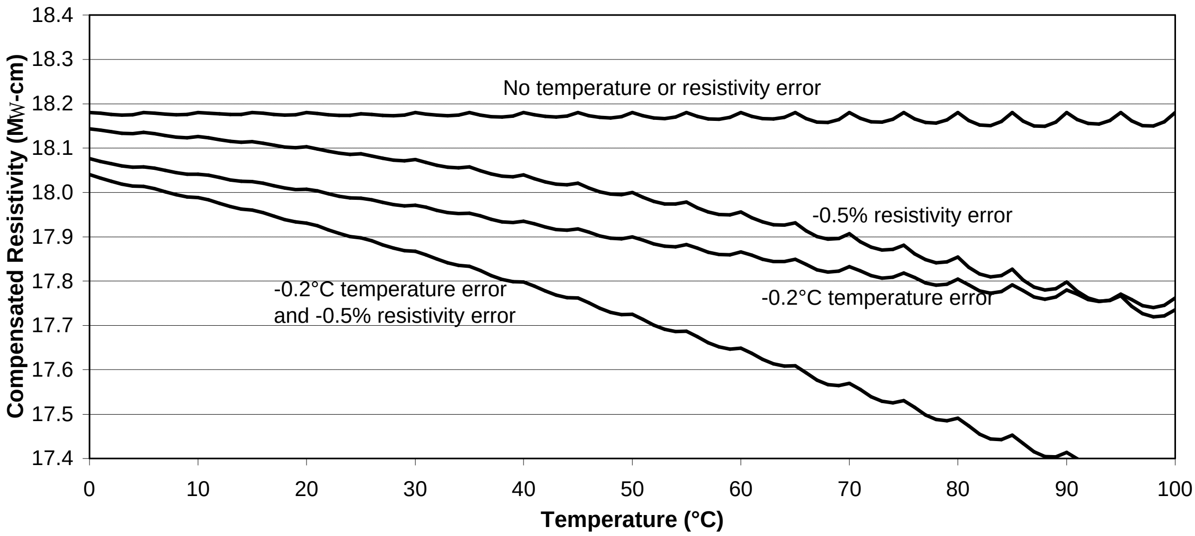
* Increase relative to UPW 55.0 nS/cm at 25°C
** Increase relative to UPW 468 nS/cm at 80°C

Figure 1. Change in Conductivity as a Function of Impurity Concentration



The ability to distinguish a temperature increase from an impurity invasion puts further emphasis on the accuracy of the measurements. Figure 2 illustrates the problems that arise from very small errors. When the temperature measurement is incorrect by as little as 0.2°C at 25°C, the decrease in temperature compensated resistivity is 1%, from 18.18 to 17.98 MΩ-cm. Yet the same temperature inaccuracy yields a decrease in compensated resistivity of 2%, from 18.18 to 17.8 MΩ-cm for 80°C UPW. If there is a small (0.5%) error in the raw resistivity, the calculated compensated resistivity errors are similar.

Figure 2. Effect of Measurement Errors on Compensated Resistivity



There are three key elements to accurate compensated resistivity measurements:

- Accurate resistivity measurement
- Accurate temperature measurement
- Accurate compensation algorithm

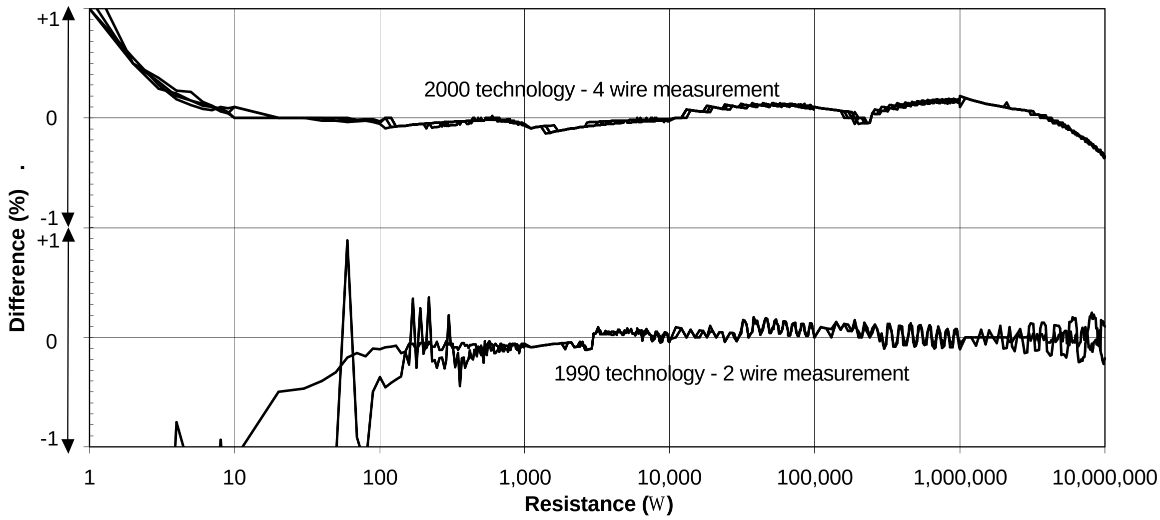
The accuracy of temperature compensation algorithms has been described elsewhere[1, 2] and is not the subject of this paper. Instead, we will focus primarily on the improvements made in resistivity and temperature measurement accuracies.

Semiconductor Requirements

Better Resistivity Accuracy

About six years ago the use of 4-wire measurements for temperature[2] was introduced as a viable means to improve temperature accuracy. State-of-the-art analyzers now use 4-wire measurements not only to make temperature measurements, but also to make resistivity measurements. This greatly improves the analyzers ability to measure very low resistances found in the measurement of high conductive fluids such as, wastewater and acids. This has resulted in an accuracy that is greatly improved, thereby reducing the limit of detection. A typical example of the performance of the new technology and its comparison to older instruments is provided in Figure 3 below. This technology had reduced the repeatability by ~5-fold and improved the accuracy across the entire dynamic range.

Figure 3. Accuracy/Repeatability of Raw Resistance Measurements

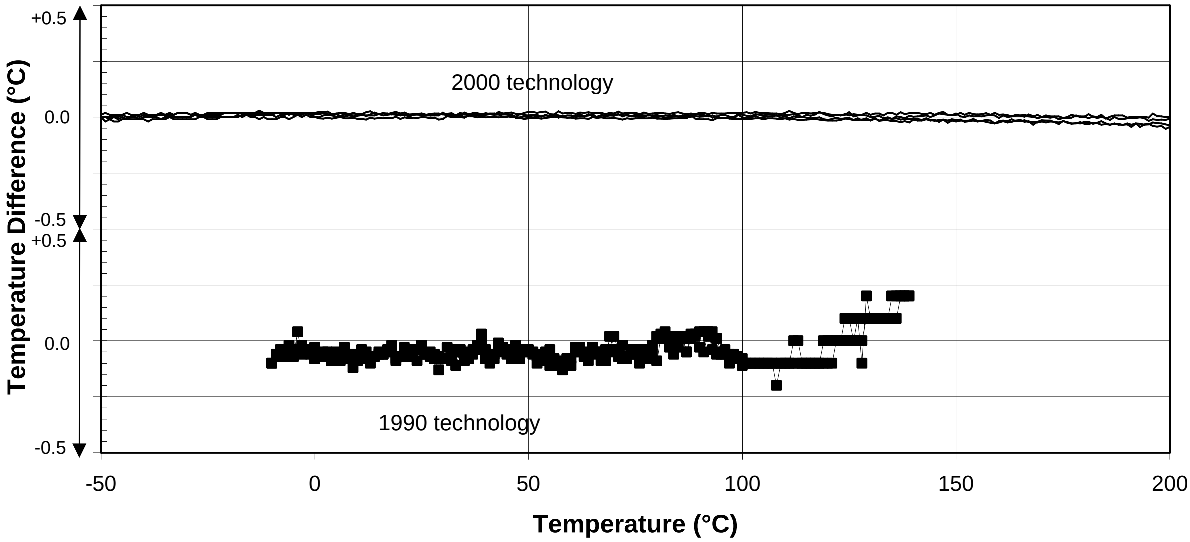


Better Temperature Accuracy

Accurate " temperature compensated" resistivity measurements depend on the instrument's ability to read the raw uncompensated resistivity and temperature of the water, while utilizing the most accurate compensation algorithm. The latter factor has been the subject of a number of reports[2-4]. When inaccurate compensated resistivity measurements are found, the most common response is to fault the measurement cell or the meter accuracy, but it is often an inaccurate temperature measurement that causes inaccurate compensated resistivity measurements.

Figure 2, illustrates that a temperature error as small as -0.2°C has a greater impact on compensated resistivity than a -0.5% resistivity error. Although -0.2°C may seem to be a significant error, on a 1000 Ω Pt RTD it represents only 0.7 Ω, or 0.07%. It is the compensated resistivity measurement's reliance on temperature that magnifies temperature errors. It is therefore important for instrument manufactures to not only improve the analyzer's ability to read resistivity, but to also address and improve the analyzer's ability to measure temperature accurately. In this area, they have made great strides. With new circuitry and calibration methods, modern resistivity analyzers have greatly reduced noise and, as shown on Figure 4, improved accuracy.

Figure 4. Accuracy/Repeatability of Temperature Measurements



These advances have lead to the ability of detecting lower and lower ionic impurities. Today, resistivity analyzers have compensated resistivity measurement accuracies of 1% at 25°C. This corresponds to 250 ppt as NaCl and 120 ppt as the acid chloride. Unfortunately, these low detection limits cannot be reproduced at elevated temperatures. The same raw measurement accuracy results in compensated measurements inaccuracies of 3.5%, which results in a detection limit of ~3.5 times greater.

Wider Dynamic Ranges

It is cost effective for facility operators to stock and inventory resistivity analyzers that have wide dynamic ranges. After all, if an UPW system operator can stock a single analyzer that can be placed anywhere in the water system, he can significantly reduce his inventory costs. But an UPW system can have a very broad range of conductivity requirements; from cold UPW to hot concentrated acid. Cold UPW (0°C) has a resistivity of ~86 MΩ-cm (0.012 µS/cm) and hot 12% HCl has a resistivity of ~1 Ω-cm (1 S/cm), this nearly spans 8 decades. Most resistivity analyzers have a typical dynamic range of 10^2-10^3 when used with one sensor, and require 3 or more sensors to span the entire range. Still, other analyzers may only work, or meet accuracy specifications, at one end of the spectrum or the other, but not both.

To meet today's requirements, instrument manufacturers have designed instruments that work accurately across the entire resistivity spectrum with fewer sensors. By combining new advances in hardware, 4-wire measurements accuracies, different measurement frequencies, and newer sampling algorithms instrument manufactures have produced instruments which have extended the dynamic range by $>10^2$ over older instruments, without negatively impacting accuracy. Modern instruments can now work accurately across the entire resistivity spectrum with as little as two sensors.

Other Improvements

Multi-Parameter Instruments

The multi-parameter concept was originally introduced back in the mid 80's. This concept permitted a single analyzer to measure different parameters, such as resistivity, conductivity, temperature, % chemical concentration, pH, flow, pressure, and tank level. But the operator had to physically change the analyzer's internal PC boards to measure the different parameters. Then, in the early 90's new advances were introduced to the multi-parameter concept with the introduction of "Smart" sensors. The Smart Sensor™ has a non-volatile random access memory (NVRAM) device attached to it. During the manufacturing process, information such as type of sensor, type of signal produced, and calibration information is written to the NVRAM. When the sensor is attached to the analyzer, this information is automatically transmitted digitally to the analyzer enabling it to select the appropriate circuitry required by the sensor. The ability of the sensor to identify itself to the analyzer, and to new analyzer circuitry that can be activated from the sensor, eliminates the need to replace internal boards. Additionally, the calibration information is processed and the raw measurement adjusted to produce a new calibrated measurement.

Recently, more information (calibration data, last calibration date, part and serial numbers) has been added to the NVRAM to facilitate more automated Quality Assurance programs.

Calibration

Today's state-of-the-art analyzers are much more complex. They can process different types of input signals, often have multiple channels and much greater dynamic ranges. It is not uncommon for modern analyzers to require up to 72 points of calibration to meet specific accuracy goals. This can be a calibration technician's worst nightmare. Fortunately, calibration devices have been introduced that provide automated calibration. These devices have internal precision resistance, voltage and frequency sources that are traceable to national standards. Much like the Smart Sensor™, the calibrator digitally transmits the precision values to the analyzer. The analyzer compares these values to the signals from its input channels and computes calibration factors (offset, slope, and sometimes 2^{nd} degree term). This whole process takes less than 2 minutes per channel and requires only one press of a button. An experienced calibration technician, performing a manual calibration, would require at least 2 hours to connect and disconnect the various signal sources required for a full calibration.

To further facilitate the calibration process, the calibrator device is capable of storing the analyzer's measurement error before and after the calibration process. This information is neatly organized into a "Certificate of Calibration" with before and after data that can be easily downloaded via the device's serial port.

Installation Improvements

Sensor wiring has always been a tedious process, but as sensors become more complex, the number of leads required to connect them to an analyzer has increased. A once tedious process can now be a lengthy process as well. It was not uncommon for wiring errors to cause false measurements, or damage to expensive sensors and analyzers.

New connector technologies have enabled instrument manufactures to design plug and play concepts into newer devices. These new robust connectors plug directly into the meter with no labor, and therefore no possibility of wiring errors. Facilitating the installation process even further, are new watertight connectors at the end of the sensors which mate with the patch cord with just a simple ¼ turn. These connectors are especially beneficial if the sensors are located in a harsh environment where water or chemicals can corrode open connections or inferior connectors. Together with the plug and play concept at the meter, these new rugged connectors have increased system reliability and dramatically reduced installation costs.

Multiple Inputs and Measurements

Microprocessor based instrumentation has been available since the 1980's. But for the most part these instruments offered only a single channel of measurement. When two channels were available, only one could be displayed at a time and only the primary channel permitted limited control capabilities. As the microprocessor's capabilities increased, more features, more inputs, more measurements, and more computations became available. The single channel meters of the 80's gave way to the dual channels of the 90's, and today, with the implementation of multiplexers and new measurement circuitries it is not uncommon to have four or more channel instruments. These meters can accept an assortment of different types of sensors such as:

- 2-electrode resistivity/conductivity
- 4-electrode resistivity/conductivity
- Temperature
- pH
- Flow
- Pressure
- Level

The list above only describes the type of sensor that can be used with the meter. In fact, with innovative programming and complex computations, today's analyzer can compute many more measurements from the above sensors. The following is a list of typical derived measurements from the sensors listed above:

- conductivity
- resistivity
- TDS
- concentration: % HCl, % H_2SO4, % NaOH
- temperature : °C, °F
- pH
- ORP
- flow rate : GPM, LPM, m^3/hr
- total flow : gal, liter, m^3
- pressure : PSI, bars, kg/cm^2, mm Hg

- tank level : gal, liter, m^3, ft of H_2O, % full

By combining readings from multiple channels, additional measurements can be computed.

- grains
- % recovery
- % rejection
- TOC reduction
- difference of 2 channels
- ratio of 2 channels

Other new features are the meter's ability to use different compensation algorithms. In today's semiconductor fab one can find many different kinds of fluids such as, high purity water, organic cleaning reagents like isopropyl alcohol (IPA), and acid and bases used in etching and glycol coolants. Each of these fluids requires distinctive compensation algorithms used for the different temperatures and concentration-effects on resistivity. Although linear correction factors such as ~2% per °C are adequate in many applications that are <0.1 MΩ-cm, the fluids previously listed require unique temperature compensation algorithms. Many of today's analyzers allow the operator to select the suitable algorithm by simply pressing a few keys.

Versatile Display

With today's multiple channel analyzers providing increasingly more measurements, a means to display all the available measurements was required. Instrument manufactures resolved this quandary by designing displays with more lines of information. Today's water system operator can program an analyzer to display up to four lines of information and choose which measurements to continuously view on the screen. Additionally, scroll keys are available that permit viewing of less pertinent, but nonetheless important, readings.

As an example, a resistivity/temperature sensor can provide a number of measurements, such as, uncompensated (raw) resistivity compensated resistivity, temperature, and TDS. The combination of 2 sensors can produce measurements derived from both, such as, % rejection, % recovery, or ratios. Because of space limitations, all readings cannot be displayed at the same time. Today's analyzers allow the operator to easily select the most relevant measurements for continuous display and other less critical measurements may be selected and viewed as required.

More and Better Control

Traditionally, instrument manufacturers provided 2 or 3 setpoints that the operator could configure to activate relays or indicate alarm conditions. But system operators require more versatility to control today's complex water systems. This requirement was met with the introduction of multi-parameter analyzers with a dozen or more setpoints that not only provide a visual alarm indication, but can also activate relays to provide more flexible controls. Additionally, modern analyzers allow programming of the setpoints to activate not only on a "high" or "low" conditions, but can also be programmed to "reset" totalized measurements or activate on a specific temperature-dependent conductivity limit.

To provide even greater system control, relays also have programming features that can be utilized. Multiparameter analyzers permit the operator to program hysteresis, delays, and normal or inverted activation on the relays. These features, together with the setpoint programmability, provide fail-safe system operation. But, because today's analyzers utilize advanced microprocessor technology, additional digital I/O lines are also provided. While digital outputs can be used to activate PLC inputs or external relays, digital inputs can be used to initiate a "reset" command which is used to zero totalized measurements. For example, if total flow is monitored to control a batch process, a digital input can be programmed to activate and reset the total flow measurement to "0" when the batch process is complete. The batch process can then be repeated as required.

Additional 0/4-20 mA analog outputs have been introduced to support the multiparameter features of the modern analyzer. While older analyzers had only 1 or 2 analog outputs, today's analyzers can have 4 or more analog outputs to accommodate the many measurements available. The use of sensors over a wide dynamic range also demands a wider range of user-selectable options, such as, inverted outputs, logarithmic outputs, and multi-ranging outputs.

Summary

The ability of UPW system fabricators to produce water with lower ionic concentrations and the demand to reduce costs, has resulted in the introduction of a new generation of resistivity analyzers. Today's resistivity analyzers are more accurate, have more inputs and outputs, are easier to operate, and control more of the water system then ever before.

Acknowledgements

The author wishes to express his gratitude to Anthony C. Bevilacqua, Ph.D. for his insight and contribution this paper. Dr. Bevilacqua presented portion of this paper at 2000 Semiconductor Pure Water and Chemicals Conference.

Biography

Victor M. Braga received a Certificate of Electronic Technology from Sylvania Technical School (now Wentworth Institute of Technology). Vic joined Thornton Inc. in 1984 and has held various positions within the company, including participation in the 770PC and 770MAX development teams, which introduced the first multi-parameter instruments and the patented Smart Sensor™ technology. He has traveled extensively providing application and instrument training support to various semiconductors manufacturers. In 1998 Vic was promoted to Technical Service Manager. In his current position, Vic leads an international group of service technicians that provide instrument calibration, repair and technical support worldwide.

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