Is There an Accurate Low-Conductivity Standard Solution?

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"Certified" low-conductivity solutions down to 10 microsiemens/cm are readily available for the calibration of conductivity/resistivity instrumentation. Can these standards be relied upon for accurate and certified calibrations? What are the practical tolerances of these standards? The surprising answers to these questions are valuable to anyone responsible for high-purity water quality. For regulated industries, the ramifications of the findings could mean the difference between being in or out of compliance.

It is suggested that an accurate calibration be defined as a calibration within 1%-2% tolerance. This tolerance was chosen because many instrument manufacturers' tolerances fall into this range. Furthermore, the United States Pharmacopeia guidelines [1] under the USP 24 Monographs require a cell calibration within a 2% tolerance. [2] Key industries affected by such issues are pharmaceutical, biotechnology, semiconductor, and power generation. This paper contains fresh experimental data, determines the accuracy of some low-conductivity standards, and evaluates the feasibility of their intended use.

Rosemount launched this study in response to an incredible amount of confusion and controversy on the part of end-users concerning high-purity conductivity calibrations and low-conductivity standards. The study was performed in cooperation with an end-user of both conductivity instrumentation and conductivity solution standards, Chiron Technologies. Chiron Technologies is a leading biotechnology firm based in San Diego, California. Chiron's Metrology Laboratory Supervisor, John Jacanin, provided independent verification of the Rosemount laboratory methods and ensured that the data, analysis, and conclusions presented in this paper are non-biased. Similarly, Southern California Edison's laboratory was contracted by Rosemount to perform an independent and formal confirmation of some low conductivity standards solutions. The experimental data from both laboratories was analyzed, and conclusions regarding both accuracy and the magnitude of error are provided. In addition, alternative calibration methods are presented.

Methods

The standard solutions tested in this study were purchased from common laboratory supply catalog companies and from specialty chemical companies. An attempt was made to purchase standard solutions directly from the National Institute of Standards and Technology (NIST); however, all of the low-conductivity standards were on indefinite backorder. The NIST customer service person was unable to provide an estimated availability date. A similar attempt to purchase NIST low-conductivity standards over a year ago yielded the same results.

Low-conductivity solutions from three sources were tested in this study. Representative samples were tested at both the Rosemount Analytical laboratory and an independent water chemistry laboratory (Southern California Edison Co., Water Technology Resources Laboratory). The majority of the solutions were tested at Rosemount. The SCE Lab findings were used to validate the Rosemount findings. Due to the nature of the sample solutions, they can only be measured once. For each low-conductivity solution standard, the stated conductivity value and tolerance was taken off the certificate of analysis that was affixed to its container.

The sealed containers of solution standards were placed in a constant temperature bath to adjust their temperature to 25.0 °C ±0.05 °C. The containers were periodically shaken to ensure proper mixing and ample time was allowed for temperature stabilization. The conductivity measurements were taken immediately after breaking the container's seal.

The solutions were measured with certified reference lab cells and conductivity bridges. The two laboratories conducted tests completely independent of one another, using their own reference cells and conductivity bridges, lab equipment, supplies, and quality systems. Coincidentally, the precise reference instruments used at each of the laboratories were of the same make and models. Both laboratories measured conductivity with the Beckman Model RC-20 Conductivity Bridge (± 0.25%) and Beckman A-Series laboratory glass conductivity standard cells (about ± 0.3 %). The total uncertainty in the laboratory measurements is less than 1%.

The reference bridges were calibrated with NIST traceable resistors of ± 0.1% tolerance. The reference cells were calibrated per the widely accepted American Society of Testing and Materials (ASTM) D1125 methodology, [3] using NIST traceable lab devices and glassware for the standard cell calibration. In the ASTM D1125 method, a known conductivity solution is freshly prepared per a detailed chemical recipe and following strict procedures. The reference sensor to be certified is placed into this known solution and its exact cell constant is determined. The certified reference cell can then be used to calibrate other test cells in side-by-side comparisons. Certified reference cells hold their calibration well with typical re-certification schedules of three months or more.

For the solutions in plastic containers with wide-mouth openings, the conductivity measurement was performed directly in the original container after rinsing the reference cell with 18 Megohm-cm water. For the glass-bottled containers, the solution standard was transferred to a meticulously cleaned and rinsed beaker that had been brought to constant temperature in the same bath. The conductivity measurement was then performed in the beaker after rinsing the reference cell with some of the sample.

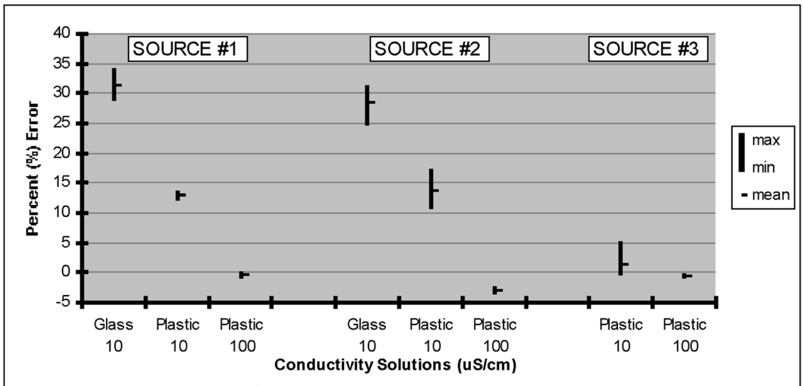

Figure 1. Range of Error for Low-Conductivity Solutions.

Results

The test results from each of the three sources are presented separately, below. The maximum, minimum, and mean percent errors are graphed in Figure 1 for each of the three sources.

Source #1

Table A presents the detailed results from testing the nominal 10 µS/cm low-conductivity standard solutions from Source #1 that were packaged in glass bottles. Eight samples from three different lots were tested. The Rosemount lab

Table A.

SOURCE # 1

All measurements at 25.0 Degrees C
Lab readings tolerance: Less than 1%

Package	Lot ID	Solution's Stated Value (µS/cm)	Solution's Stated Tolerance	Error (µS/cm)	Percent % Error	Sample #	Rosemount Lab (µS/cm)	SCE Lab (µS/cm)
Glass	a	10.0	± 0.25 (µS/cm)	3.22	32	1	13.22	n/a
Glass	a	10.0	± 0.25 (µS/cm)	3.31	33	2	13.31	n/a
Glass	b	10.1	± 0.25 (µS/cm)	2.90	29	3	13.00	n/a
Glass	b	10.1	± 0.25 (µS/cm)	3.13	31	4	n/a	13.23
Glass	b	10.1	± 0.25 (µS/cm)	3.14	31	5	n/a	13.24
Glass	b	10.1	± 0.25 (µS/cm)	3.02	30	6	n/a	13.12
Glass	c	10.2	± 0.25 (µS/cm)	3.43	34	7	n/a	13.63
Glass	c	10.2	± 0.25 (µS/cm)	3.23	32	8	n/a	13.43
					mean = 31.41		mean = 13.18	mean= 13.33
							std.dev.= 0.16	std.dev.= 0.20

tested three of the samples and determined the mean conductivity to be 13.18 µS/cm with a standard deviation of 0.16 µS/cm. The SCE lab tested five of the samples and determined the mean conductivity to be 13.33 µS/cm with a standard deviation of 0.20 µS/cm. The errors ranged between 29 to 34% with a mean of 31.41% (see Figure 1).

Table B presents the detailed results from testing the nominal 10 µS/cm low-conductivity standard solutions from

SOURCE # 1

All measurements at 25.0 Degrees C
Lab readings tolerance: Less than 1%

Package	Lot ID	Solution's Stated Value (µS/cm)	Solution's Stated Tolerance	Error (µS/cm)	Percent % Error	Sample #	Rosemount Lab (µS/cm)	SCE Lab (µS/cm)
Plastic	d	9.7	± 0.25 (µS/cm)	1.28	13.2	1	10.98	n/a
Plastic	d	9.7	± 0.25 (µS/cm)	1.20	12.4	2	10.90	n/a
Plastic	d	9.7	± 0.25 (µS/cm)	1.30	13.4	3	11.00	n/a
					mean = 12.99		mean = 10.96 std.dev.= 0.05	

Table B.

Source #1 that were packaged in plastic containers. Three samples from one lot were tested. The Rosemount lab tested the three samples and determined the mean conductivity to be 10.96 µS/cm with a standard deviation of 0.05 µS/cm. The errors ranged between 12 to 13% with a mean of 12.99% (see Figure 1).

Table C presents the detailed results from testing the nominal 100 µS/cm low-conductivity standard solutions from

SOURCE # 1

All measurements at 25.0 Degrees C
Lab readings tolerance: Less than 1%

Package	Lot ID	Solution's Stated Value (µS/cm)	Solution's Stated Tolerance	Error (µS/cm)	Percent % Error	Sample #	Rosemount Lab (µS/cm)	SCE Lab (µS/cm)
Plastic	e	99.0	± 0.25 %	-0.6	-0.6	1	98.37	n/a
Plastic	e	99.0	± 0.25 %	-0.3	-0.3	2	98.75	n/a
Plastic	e	99.0	± 0.25 %	-0.1	-0.1	3	98.91	n/a
					mean = -0.33		mean = 98.68 std.dev.= 0.28	

Table C.

Source #1 that were packaged in plastic containers. Three samples from one lot were tested. The Rosemount lab tested the three samples and determined the mean conductivity to be 98.68 µS/cm with a standard deviation of 0.28 µS/cm. The errors ranged between -0.1 to -0.6% with a mean of –0.33% (see Figure 1).

Source #2

The solution standards from Source #2 had identical packaging to the solution standards from Source #1. The authors suspect that the solutions are made by the same manufacturer and different vendor names are applied to the labels.

Table D presents the detailed results from testing the nominal 10 µS/cm low-conductivity standard solutions from Source #2 that were packaged in glass bottles. Three samples from two lots were tested. The Rosemount lab tested two of the samples and determined the mean conductivity to be 12.85 µS/cm with a standard deviation of 0.66 µS/cm. The SCE lab tested one of the samples and determined the conductivity to be 13.45 µS/cm. The errors ranged between 25 to 31% with a mean of 28.29% (see Figure 1).

Table E presents the detailed results from testing the nominal 10 µS/cm low-conductivity standard solutions from Source #2 that were packaged in plastic containers. Seven samples from one lot were tested. The Rosemount lab tested five of the samples and determined the mean conductivity to be 11.09 µS/cm with a standard deviation of

SOURCE # 2						All measurements at 25.0 Degrees C Lab readings tolerance: Less than 1%		
Package	Lot ID	Solution's Stated Value (μS/cm)	Solution's Stated Tolerance	Error (μS/cm)	Percent % Error	Sample #	Rosemount Lab (μS/cm)	SCE Lab (μS/cm)
Glass	f	9.9	± 0.25 (μS/cm)	2.5	25	1	12.38	n/a
Glass	n	10.3	± 0.25 (μS/cm)	3.2	31	2	n/a	13.45
Glass	n	10.3	± 0.25 (μS/cm)	3.0	29	3	13.31	n/a
					mean = 28.29		mean = 12.85	mean = 13.45
							std.dev.= 0.66	std.dev.= n/a

Table D.

0.17 μS/cm. The SCE lab tested two of the samples and determined the conductivity to be 11.25 μS/cm with a standard deviation of 0.35 μS/cm. The errors ranged between 11 to 17% with a mean of 13.62% (see Figure 1).

Table F presents the detailed results from testing the nominal 100 μS/cm low-conductivity standard solutions from

SOURCE # 2						All measurements at 25.0 Degrees C Lab readings tolerance: Less than 1%		
Package	Lot ID	Solution's Stated Value (μS/cm)	Solution's Stated Tolerance	Error (μS/cm)	Percent % Error	Sample #	Rosemount Lab (μS/cm)	SCE Lab (μS/cm)
Plastic	g	9.8	± 0.25 (μS/cm)	1.56	16	1	11.36	n/a
Plastic	g	9.8	± 0.25 (μS/cm)	1.32	13	2	11.12	n/a
Plastic	g	9.8	± 0.25 (μS/cm)	1.25	13	3	11.05	n/a
Plastic	g	9.8	± 0.25 (μS/cm)	1.21	12	4	11.01	n/a
Plastic	g	9.8	± 0.25 (μS/cm)	1.10	11	5	10.90	n/a
Plastic	g	9.8	± 0.25 (μS/cm)	1.70	17	6	n/a	11.50
Plastic	g	9.8	± 0.25 (μS/cm)	1.20	12	7	n/a	11.00
					mean = 13.62		mean = 11.09	mean = 11.25
							std.dev.= 0.17	std.dev.= 0.35

Table E.

Source #2 that were packaged in plastic containers. Three samples from one lot were tested. The Rosemount lab tested the three samples and determined the mean conductivity to be 98.68 μS/cm with a standard deviation of 0.28 μS/cm. The errors ranged between -2.7 to -3.3% a mean of –2.97% (see Figure 1).

Table G presents the detailed results from testing the nominal 10 μS/cm low-conductivity standard solutions from

SOURCE # 2						All measurements at 25.0 Degrees C Lab readings tolerance: Less than 1%		
Package	Lot ID	Solution's Stated Value (μS/cm)	Solution's Stated Tolerance	Error (μS/cm)	Percent % Error	Sample #	Rosemount Lab (μS/cm)	SCE Lab (μS/cm)
Plastic	h	101.7	± 0.25 %	-3.3	-3.3	1	98.37	n/a
Plastic	h	101.7	± 0.25 %	-3.0	-2.9	2	98.75	n/a
Plastic	h	101.7	± 0.25 %	-2.8	-2.7	3	98.91	n/a
					mean = -2.97		mean = 98.68	
							std.dev.= 0.28	

Table F.

SOURCE # 3

All measurements at 25.0 Degrees C
Lab readings tolerance: Less than 1%

Package	Lot ID	Solution's Stated Value (uS/cm)	Solution's Stated Tolerance	Error (μS/cm)	Percent % Error	Sample #	Rosemount Lab (μS/cm)	SCE Lab (μS/cm)
Plastic	k	10.0	± 0.03 (μS/cm)	-0.10	-1.0	1	9.896	n/a
Plastic	k	10.0	± 0.03 (μS/cm)	-0.11	-1.1	2	9.895	n/a
Plastic	k	10.0	± 0.03 (μS/cm)	-0.10	-1.0	3	9.898	n/a
Plastic	k	10.0	± 0.03 (μS/cm)	-0.09	-0.9	4	9.906	n/a
Plastic	k	10.0	± 0.03 (μS/cm)	0.50	5.0	5*	10.50	n/a
Plastic	k	10.0	± 0.03 (μS/cm)	0.37	3.7	6*	n/a	10.37
Plastic	p	10.0	± 0.03 (μS/cm)	0.31	3.1	7	10.31	n/a
Plastic	p	10.0	± 0.03 (μS/cm)	0.25	2.5	8	10.25	n/a
					mean = 1.28		mean = 10.09	mean = 10.37
							std.dev.= 0.25	

*Tested at a later point in time than samples 1-4.

Table G.

Source #3 that were packaged in plastic containers. Eight samples from two lots were tested. The Rosemount lab tested seven of the samples and determined the mean conductivity to be 10.09 μS/cm with a standard deviation of 0.25 μS/cm. The SCE lab tested one of the samples and determined the conductivity to be 10.37 μS/cm. The errors ranged between -0.9 to +5.0% with a mean of 1.28% (see Figure 1).

Table H presents the detailed results from testing the nominal 100 μS/cm low-conductivity standard solutions from Source #3 that were packaged in plastic containers. Four samples from one lot were tested. The Rosemount lab tested the four samples and determined the mean conductivity to be 99.38 μS/cm with a standard deviation of 0.07 μS/cm. The errors ranged between -0.5 to -0.7% with a mean of -0.62% (see Figure 1).

Conclusions

The conclusions presented are related to the conductivity range of the solution and the packaging of the solution.

Conductivity Range of the Solution

Nominal 10 μS/cm Solutions:

The experimental data indicated substantial differences between the stated conductivities and the actual conductivities measured. The errors ranged from -0.9% to +34% with the majority in excess of 11%. Only four samples from one vendor were verified to be within 2%. Unfortunately, those results were not consistent even within the same batch from that particular source with errors up to 5%. Clearly, the magnitudes of the errors are

SOURCE # 3

All measurements at 25.0 Degrees C
Lab readings tolerance: Less than 1%

Package	Lot ID	Solution's Stated Value (μS/cm)	Solution's Stated Tolerance	Error (μS/cm)	Percent % Error	Sample #	Rosemount Lab (μS/cm)	SCE Lab (μS/cm)
Plastic	m	100.0	± 0.1(μS/cm)	-0.7	-0.65	1	99.35	n/a
Plastic	m	100.0	± 0.1(μS/cm)	-0.5	-0.53	2	99.47	n/a
Plastic	m	100.0	± 0.1(μS/cm)	-0.7	-0.70	3	99.30	n/a
Plastic	m	100.0	± 0.1(μS/cm)	-0.6	-0.61	4	99.39	n/a
					mean = -0.62		mean = 99.38	
							std.dev.= 0.07	

Table H.

unacceptable, and these 10 µS/cm conductivity solutions can not be used for an accurate calibration.

Nominal 100 µS/cm Solutions:

The errors were much smaller for the nominal 100 µS/cm conductivity solutions than were experienced with the nominal 10 µS/cm solutions. The errors ranged from -0.1% to -3.3% with the majority less than 1%. Only three samples from one vendor were verified to be outside 2%. With these limited errors, a carefully selected 100 µS/cm conductivity solution can be used for an accurate calibration (within 2%). Although a reasonable standard, the 100 µS/cm solution has limited applications because it is out of the range of measurement for many conductivity instruments that are designed to measure high-purity and ultra-pure water.

Packaging of the Solution

The nominal 10 µS/cm conductivity solutions packaged in glass bottles had significantly higher measured conductivities than their counterparts packaged in plastic containers. The glass-bottled solutions had differences in excess of 25%. The majority of the plastic container solutions had differences in excess of 11%. The 10 µS/cm standards as a whole were found to be unacceptable, but the solutions packaged in glass bottles were of the highest concern.

Discussion

The authors' position and recommendations regarding low-conductivity standards are presented below. In addition, the stated tolerances of the low-conductivity standards are debated and alternative calibration methods are offered.

Position Statement

This study's findings support the published recommendations of the American Society for Testing and Materials (ASTM). The ASTM Standard Test Method D5391 [4] states: "…due to the high uncertainties of atmospheric and container surface contamination, direct cell calibration with standard solutions *below 100 µS/cm* is not recommended." The key words in this warning are "below 100 µS/cm," suggesting the 100 µS/cm solution as an acceptable standard value. The experimental data supports this statement since the majority of the 100 µS/cm conductivity solutions tested were found to be accurate (with 2%). However, the intended ASTM warning can be fully appreciated by the data presented on the 10 µS/cm conductivity solutions.

The surprisingly excessive error in the 10 µS/cm solutions is a clear illustration of the ASTM's concern. **The authors of this study caution against the use of 10 µS/cm or similar low-value conductivity solutions.** Although opening a standard solution and exposing it to carbon dioxide is the most commonly recognized form of contamination, it is suspected that it is only a minor factor. The container surface contamination, sample handling, manufacturer's quality control, and container head-space are suggested as the major factors in a solution standard's out of the bottle conductivity.

Ramification of Findings

The fact that 10 µS/cm solutions have been found to have positive errors in excess of 30% has far-reaching ramifications related to process control and compliance. The following scenario is presented to illustrate this point. The water quality limit for pharmaceutical purified water under USP 24 requirements is 1.3 µS/cm at 25 °C. A typical action limit for this type of water system is 1.0 µS/cm. Assume the conductivity instrument used to control this process was calibrated with one of the glass-bottled 10 µS/cm standard measured in this study (maximum error found was 34%). The water quality would be out-of-compliance at 1.34 µS/cm before the instrument alerted the operator with a reading of 1.0 µS/cm. This out of compliance incident was caused by forcing the instrument reading to agree with the solution's stated value of 10 µS/cm when the solution was actually 13.4 µS/cm. Caution must be exerted because many conductivity instruments will simply accept any entered value. Fortunately, some conductivity instruments on the market include a special feature to warn the user if a serious mis-calibration, like the one described above, is attempted.

Stated Tolerances

Another area of concern with the low conductivity solution standards is their published tolerances. The tolerance on the nominal 10 µS/cm solutions from Source #3 is listed as ±0.03 µS/cm (0.3%), and the tolerances for Sources #1 and #2 are listed as ± 0.25 µS/cm (2.5%).

The 10.00 µS/cm solution's stated tolerance of 0.3% seems unlikely since it is lower than the expected propagation of error for precision conductivity laboratory cells and bridges.[5] If production conductivity instrumentation were used instead of primary lab instrumentation, then the expected tolerance would be even higher. For all of the preceding reasons, the 0.25% tolerance published on the 100 µS/cm solutions from Sources #1 and #2 are also in question.

More importantly, there is no provision for errors due to sample handling. The verified solution is being exposed to air, being placed into a container that could possibly leach contaminants, and then is stored for an undisclosed period of time before use. The typical end-user is most interested in the tolerance that they can expect when they take the solution out of the container to use it, rather than what the tolerance might have been when it went into the container.

The tolerance published on the 10 µS/cm solutions from

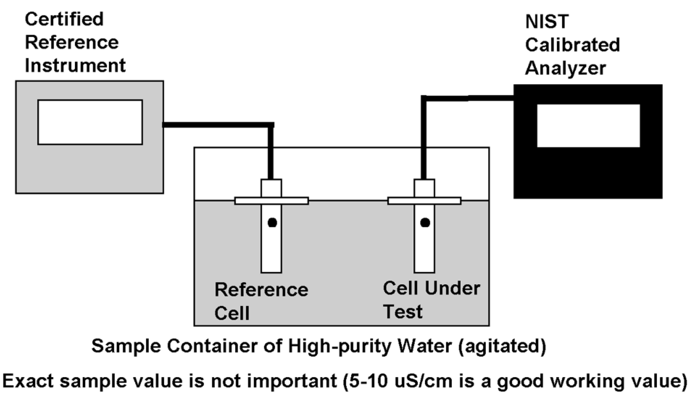

Figure 2 . Cell or Loop Calibration Set-up for a Laboratory.

Source #1 and Source #2 is of concern for a different reason. The tolerance was ± 0.25 μS/cm(2.5%). Although this tolerance seems more reasonable than the 0.3% tolerance previously mentioned, it is admittedly outside of the 2% accuracy range. Recall that the data in this study showed that these solutions could not be verified to be within this range. The USP 24 specifications [2] require the sensor's cell constant to be determined within 2%, so these solutions are unacceptable for calibration use even if they were to meet published specifications.

Alternative Calibration Method

Is there an alternative method of calibration to the use of low conductivity standard solutions? Yes, there are published calibration methods for low conductivity cells that have been widely used for many years.

One such method is detailed in ASTM Method D1125: *Standard Methods of Test for Electrical Conductivity of Water.* [3] This ASTM method describes a calibration procedure based on a comparison with a calibrated conductivity cell. Figure 2 illustrates a laboratory set-up using this approach. The cell (or loop) under test is compared against a certified reference analyzer and cell. Both cells are placed in the same sample and agitated. The exact conductivity and temperature of the water is not critical, because both cells are exposed to the same conditions at the same time. The exact cell constant of the test cell can be derived from the difference in conductivity readings between the loops. For loop calibration, the test loop's conductivity reading can simply be standardized to that of the reference loop.

Similarly, the comparison of the test instrument against a certified reference instrument can be performed without taking the test instrument out of the process. This method requires the availability of a sample tap near the test cell and specialized conductivity reference instruments. Portable conductivity validation instruments are commercially available for the purpose of on-line verification testing. Using this method, a certified reference flow-through cell is connected to the sample tap. The process water is passed directly from the sample tap through lined tubing to the flow-through cell and then to drain (see Figure 3). The process water is, therefore, isolated from interferences. The conductivity readings from the reference instrument and the unit under test can then be analyzed to determine the test sensor's cell constant or to calibrate the loop.

Call to Action

Manufacturers of low-conductivity solutions (particularly 10 μS/cm) are challenged to 1) rationalize their published specifications, 2) prove through an independent analysis that they meet their published specifications after distribution and storage, 3) provide "use by" dates if the product is found to degrade in storage, and 4) improve the product or remove it from the market if significant deficiencies are found.

End-users of low-conductivity solution standards are called upon to protect themselves through prudent investigation of these solutions. Representative samples of these solutions should be analyzed by an independent water chemistry laboratory. End-users should ensure that the independent laboratory follows a sophisticated methodology

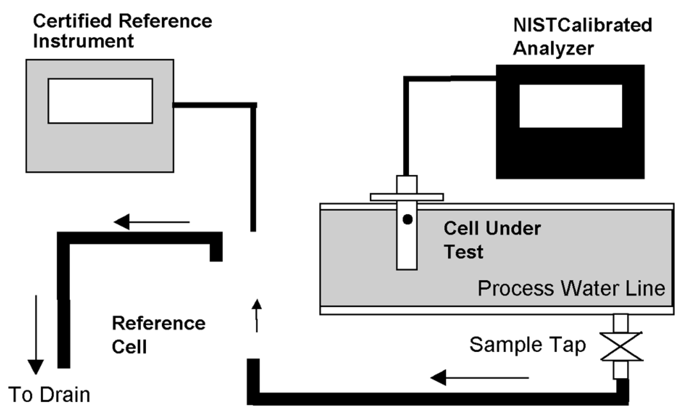

Figure 3. On-line Verification Set-up.

such as ASTM D1125 and uses NIST certified lab analyzers and cells. With the majority of reported errors on 10 μS/cm solutions in the range of 11-34%, it is crucial to ensure these solutions are not adversely impacting the user's calibration program, process control, and/or regulatory compliance.

The ASTM Method D1125 (3) is recommended to those end-users that find uncorrectable deficiencies in their low-conductivity solutions calibration program. It is designed to eliminate the concerns and uncertainties related to the use of low conductivity solutions. If the end-user does not have the resources to use and maintain the ASTM D1153 method, it is recommended that the conductivity instrumentation calibrations are contracted out to the instrument manufacturer or a qualified calibration laboratory. Just as with the solution vendors, the calibration laboratory's methods, quality control, and documentation should be thoroughly investigated.

Acknowledgements
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References

1. The United States Pharmacopeial Convention, Inc. (USP) is a legally recognized organization of representatives from colleges, and national and state organizations of medicine and pharmacy. The USP develops and publishes legally recognized standards and testing methods for the pharmaceutical industry. The guidelines are published in a document titled USP 24. The most recent version became effective in January 2000.
2. "<645> Water conductivity," USP 23 Monographs: Water for Injection & USP Purified Water, Fifth Supplement USP-NF, Sept. 15, 1996, pp. 3464-3467.
3. "Standard Test Method for Electrical Conductivity and Resistivity of Water ," Annual Book of ASTM Standards, Designation: D1125, American Society for Testing and Materials, Philadelphia, PA (December 1995).
4. "Standard Test Method for Electrical Conductivity and Resistivity of a Flowing High Purity Water Sample," Annual Book of ASTM Standards, Designation: D5391, American Society for Testing and Materials, Philadelphia, PA (June 1993).
5. Sample propagation of error calculation for a precision laboratory cell and conductivity bridge:
   Cell's relative uncertainty is 0.33% and the bridge's relative uncertainty is 0.25%:

$$\text{Propagated Relative Error} = \sqrt{(0.33\%)^2 + (0.25\%)^2} = 0.39\%$$

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