

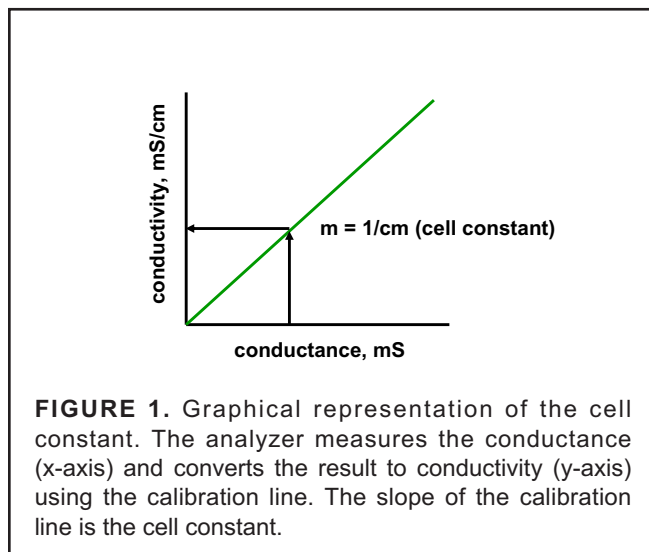
CALIBRATING TOROIDAL CONDUCTIVITY SENSORS

BACKGROUND

A toroidal conductivity sensor consists of two wire wound toroids encased in a plastic body. When an alternating voltage is applied to one of the toroids, it induces an ionic current in the solution surrounding the sensor. The ionic current, in turn, induces an electronic current in the second toroid, which the analyzer measures. The current in the second toroid depends on the number of turns of wire on each toroid and on the conductance of the solution. The conductance depends on the total ion concentration (the conductivity) and the area and length of solution the ionic current flows through. The physical dimensions of the sensor determine the dimensions of the current path.

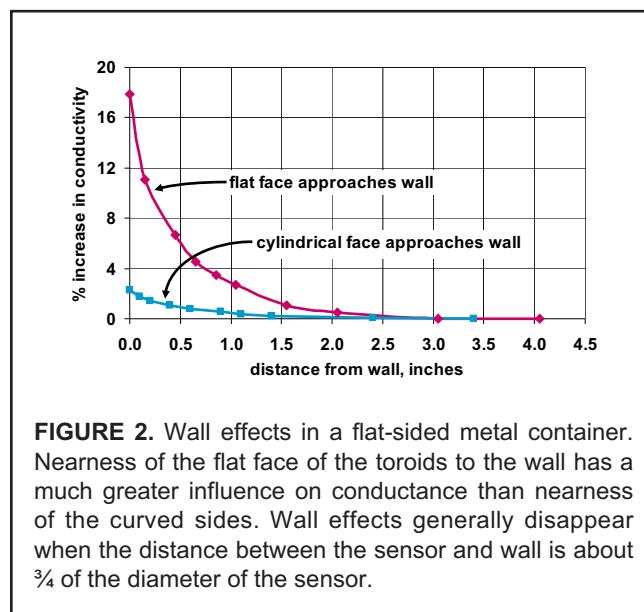
Because the purpose of the measurement is to get information about the total concentration of ions in solution, i.e., the conductivity, the effect of sensor dimensions and windings has to be accounted for. The correction factor is the cell constant. Conductivity is equal to the measured conductance multiplied by the cell constant.

The cell constant is simply a calibration factor that converts conductance (measured in mS) to conductivity (measured in mS/cm). The units of cell constant are 1/cm. Figure 1 illustrates the relationship. Note that the slope of the calibration line is the cell constant.



The cell constant is also influenced by the nearness of the vessel walls to the sensor, the so-called wall effect. Conductive and non-conductive walls have opposite effects. Metal walls increase the amount of induced current, which leads to an apparent increase in

conductance and a corresponding decrease in cell constant. Plastic walls have the opposite effect. As the Figure 2 shows, the magnitude of the wall effect also depends on the orientation of the sensor with respect to the walls.



Because nearly every installation involves some degree of wall effect, there is little value in calibrating toroidal sensors at the factory. Instead, they must be calibrated in the field. Typically, the sensor is calibrated in place in the process piping.

CALIBRATION METHODS

There are two basic ways to calibrate a toroidal sensor: against a standard solution or against a referee meter and sensor. A referee meter and sensor is an instrument that has been previously calibrated and is known to be accurate and reliable. The referee instrument can be used to perform either an in-process or a grab sample calibration. In-process calibration involves connecting the process and referee sensors in series and measuring the conductivity of the process liquid simultaneously. Grab sample calibration involves taking a sample of the process liquid and measuring its conductivity in the laboratory or shop using the referee instrument. No matter which calibration method is used, the analyzer automatically calculates the cell constant once the known conductivity is entered.

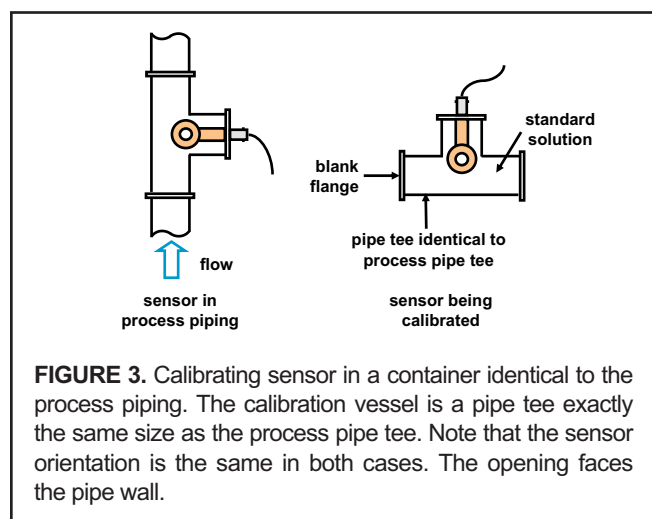
Calibration against a standard solution

Calibration against a standard solution requires removing the sensor from the process piping. It is practical only if wall effects are absent or the sensor can be calibrated in a container identical to the process piping. The latter requirement ensures that wall effects during calibration, which are incorporated into the cell constant, will be exactly the same as the wall effects when the sensor is in service.

Toroidal conductivity sensors are normally used to measure liquids having fairly high conductivity, so standard solutions having similarly high conductivity are generally most suitable for calibration. Standard solutions having conductivity as high as 200,000 uS/cm (at 25°C) are commercially available. High conductivity standards can also be prepared in the laboratory following procedures in ASTM D1125 or Standard Methods 2510. International Electrotechnical Commission Standard 746-3, Appendix B, Tables BI, BII, and BIII also contains procedures for preparing high conductivity standards.

The calibration procedure is fairly straightforward. Immerse the rinsed sensor in the standard solution and adjust the analyzer reading to match the conductivity of the standard. For an accurate calibration several precautions are necessary.

- If wall effects are absent in the installation use a sufficiently large container to ensure that wall effects are also absent during calibration. To check for wall effects, fill the container with solution and place the sensor in the center submerged at least $\frac{3}{4}$ of the way up the stem. Note the reading. Then move the sensor small distances from the center and note the reading in each position. The readings should not change.
- If wall effects are present, be sure the vessel used for calibration has exactly the same dimensions as the process piping. Also, ensure that the orientation of the sensor with respect to the piping is exactly the same in the process and calibration vessels. See Figure 3.

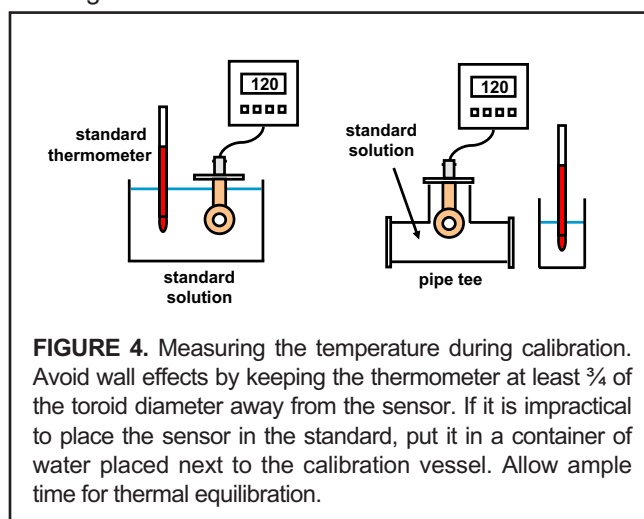


- Ideally, the conductivity of the standard should be close to the middle of the range over which the sensor

will be used. However, toroidal conductivity sensors have good linearity, so any standard greater than about 5000 uS/cm at 25°C is adequate.

- Turn off automatic temperature compensation in the analyzer. Almost all process conductivity analyzers feature automatic temperature compensation in which the analyzer applies one of several temperature correction algorithms to convert the measured conductivity to the value at a reference temperature, typically 25°C. Although temperature correction algorithms are useful for routine measurements, they should not be used during calibration. There are two reasons. First, no temperature correction algorithm is perfect. If the assumptions behind the algorithm do not perfectly fit the solution being measured, the temperature-corrected conductivity will be in error. Second, if the temperature measurement itself is in error, the corrected conductivity will be in error. The purpose of calibrating the sensor is to determine the cell constant. To minimize the error in the cell constant, all sources of avoidable error i.e., temperature compensation, should be eliminated.

- Use a standard for which the conductivity as a function of temperature is known. The information will usually be on the bottle label. If not, it can often be calculated if the identity and concentration of the electrolyte is known.
- Use a good quality calibrated thermometer to measure the temperature of the standard solution. The thermometer error should be less than $\pm 0.1^\circ\text{C}$. Allow adequate time for the solution and sensor to reach thermal equilibrium. If the sensor is being calibrated in an open beaker, keep the thermometer far enough away from the sensor so it does not introduce wall effects. If the sensor is being calibrated in a pipe tee or similar vessel, it will probably be impractical to place the thermometer in the standard solution. Instead, put the thermometer in a beaker of water placed next to the calibration vessel. Let both come to thermal equilibrium with the ambient air before continuing the calibration. See Figure 4



- If the conductivity of the standard is known only at 25°C, the temperature of the solution must be adjusted to $25.0 \pm 0.1^\circ\text{C}$ before completing the cali-

bration. Use a water bath to adjust the temperature. Do not use a hot plate. Use a good quality thermometer to measure the temperature. If wall effects or the shape of the vessel make it impossible to put the thermometer in the standard solution, put it in the water bath instead. Keep the thermometer as close as possible to the vessel containing the standard. Allow plenty of time for thermal equilibration.

- Follow good laboratory practice. Rinse the container and sensor at least twice with standard. Discard all rinses and the standard after it has been used to calibrate the sensor. Do not attempt to recycle used standard. Keep the bottle of standard tightly capped unless you are pouring from it.
- Be sure air bubbles are not adhering to the sensor. An air bubble trapped in the toroid opening has a particularly severe effect on the reading. Air bubbles are normally easy to see. However, if the sensor is not visible when it is in the calibration vessel, check for bubbles by noting any change in the reading caused by repeatedly inserting and removing the sensor from the vessel. If repeated readings agree, trapped bubbles are absent. If they do not agree, bubbles are present.

Calibration against a referee – in-process

This method involves connecting the process and referee sensors in series and allowing the process liquid to flow through both. The process sensor is calibrated by adjusting the process analyzer reading to match the conductivity measured by the referee instrument. Figure 5 shows the arrangement.

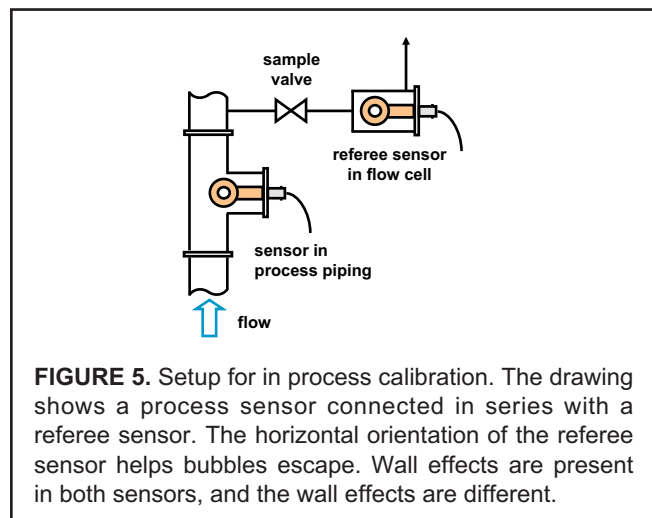


Figure 5 suggests two reasons why in-process calibration using toroidal sensors is often impractical. First, in-process calibration requires the referee sensor be installed in a flow cell, making it highly likely that wall effects will be present. For the referee sensor to accurately measure conductivity, the wall effects in the flow cell must be incorporated into the referee sensor cell constant. Thus, the referee sensor must be calibrated in the flow cell, ideally using a standard solution. The second problem involves sample handling. Toroidal conductivity measurements

are often made in corrosive or dirty solutions. In-process calibration involves testing a sidestream sample, which introduces the problem of handling and disposing of the sample stream leaving the referee flow cell.

For a successful calibration, several precautions are necessary.

- If possible, adjust the conductivity of the process liquid so that it is near the midpoint of the operating range. If this is not possible, adjust the conductivity so that it is at least 5000 uS/cm.
- Orient the referee sensor so that air bubbles always have an easy escape path and cannot get trapped. Tap and hold the flow cell in different positions to allow bubbles to escape.
- Turn off automatic temperature compensation in the analyzer. Almost all process conductivity analyzers feature automatic temperature compensation in which the analyzer applies one of several temperature correction algorithms to convert the measured conductivity to the value at a reference temperature, typically 25°C. Although temperature correction algorithms are useful for routine measurements, they should not be used during calibration. There are two reasons. First, no temperature correction algorithm is perfect. If the assumptions behind the algorithm do not perfectly fit the solution being measured, the temperature-corrected conductivity will be in error. Second, if the temperature measurement itself is in error, the corrected conductivity will be in error. The purpose of calibrating the sensor is to determine the cell constant. To minimize the error in the cell constant, all sources of avoidable error i.e., temperature compensation should be eliminated.
- Keep tubing runs between the sensors short and adjust the sample flow to as high a rate as possible. Short tubing runs and high flow ensure that the temperature of the liquid does not change as it flows from one sensor to another. If the process temperature is appreciably different from ambient, high flow may not be enough to keep temperature constant. In this case, it will be necessary to use automatic temperature correction. See the section below for precautions when using automatic temperature compensation.
- Wait until readings are stable before starting the calibration.

Calibration against a referee – grab sample

This method is useful when calibration against a standard is impractical or when in-process calibration is not feasible because the sample is hot, corrosive, or dirty, making handling the waste stream from the referee sensor difficult. The method involves taking a sample of the process liquid, measuring its conductivity using a referee instrument, and adjusting the reading from the process analyzer to match the measured conductivity.

For a successful calibration, several precautions are necessary.

- If possible, adjust the conductivity of the process

liquid so that it is near the midpoint of the operating range. If this is not possible, adjust the conductivity so that it is at least 5000 uS/cm.

- Take the sample from a point as close to the process sensor as possible. Be sure the sample is representative of what the sensor is measuring.
- Keep temperature compensation turned on. There is likely to be a lag time between sampling and analysis, so temperature is likely to change. Therefore, temperature compensation, although imperfect, must be used to compensate for temperature changes. Be sure the referee and process instruments are using the same temperature correction algorithm, which will most likely be linear slope. Because most analyzers allow the user to adjust the slope value, confirm that the process and referee instruments are using the same value. If the referee instrument does not have automatic linear slope correction, calculate the conductivity at 25°C using the equation:

$$C_{25} = \frac{C_t}{1+a(t-25)}$$

Where C_{25} is the conductivity at 25°C, C_t is the conductivity at $t^\circ\text{C}$, and a is the temperature coefficient expressed as a decimal fraction.

- Confirm that the temperature measurements in both the process and referee instruments are accurate, ideally to within $\pm 0.5^\circ\text{C}$.
- Most likely the conductivity of process liquid will be high, so it will be appropriate to measure its conductivity using a referee toroidal sensor and analyzer. Observe the following precautions.
 - Avoid wall effects. Calibrate the referee instrument against a standard solution in a vessel large enough to eliminate wall effects. Similarly, measure the conductivity of the grab sample in a large container. Wall effects generally disappear when the distance between the sensor and the wall is about $\frac{3}{4}$ of the diameter of the sensor.
 - Rinse the container and sensor at least twice with sample.
 - Be sure air bubbles are not trapped on the referee sensor or in the toroid opening.
 - Test the sample as quickly as possible after collection. The less the sample temperature changes from the temperature of the process liquid, the smaller will be the temperature correction error.
 - Usually, automatic temperature correction is based on the temperature measured by an RTD or thermistor embedded in the plastic body of the sensor. Plastic is a poor conductor of heat, so it may take 30 minutes or longer for temperature equilibration. During that time, the grab sample temperature will likely change. Because the temperature coefficient is usually a function of temperature, any difference between the process temperature and the temperature at which the grab sample is measured has the potential to increase the error in the temperature-corrected conductivity. To minimize the error,

preheat or pre-cool the referee sensor by placing it in water having approximately the same temperature of the process liquid.

- Wait until readings are stable before starting the calibration.
- Be sure to compensate for process conductivity changes that might have occurred while the grab sample was being tested. Newer Rosemount Analytical conductivity analyzers (Models 1056, 1066, and 56) do this automatically. They save the value of the process conductivity at the time the sample was taken and use that value to calculate the new cell constant when the user enters the result of the grab sample test. Older analyzers do not remember the process conductivity value. Therefore, the user must enter a value adjusted by an amount proportional to the change in the process conductivity. For example, suppose the process conductivity is 81.0 mS/cm when the sample is taken and 81.5 mS/cm when the test result is entered. If the grab sample conductivity is 81.9 mS/cm, the user should enter $(81.5/81.0)(81.9)$ or 82.4 mS/cm.

Calibration – percent concentration

Toroidal sensors are often used to measure the concentration of aqueous solutions, for example the acid or caustic used to regenerate ion-exchange demineralizers. In these applications, the sensors are conveniently calibrated against the results of a chemical analysis made on the grab sample

For a successful calibration, several precautions are necessary.

- If possible, adjust the concentration so that it is above the midpoint of the operating range. Do not calibrate the sensor when the conductivity is near the low end of the operating range.
- Take the sample from a point as close to the process sensor as possible. Be sure the sample is representative of what the sensor is measuring.
- Configure the analyzer to read percent concentration using either one of the predetermined curves in the analyzer or a custom curve calculated from data entered by the user.
- Wait until readings are stable before starting the calibration.
- Be sure to compensate for process changes that might have occurred while the grab sample was being tested. Newer Rosemount Analytical conductivity analyzers (Models 1056, 1066, and 56) do this automatically. They save the concentration value at the time the sample was taken and use that value to calculate the new cell constant when the user enters the result of the grab sample test. Older analyzers do not remember the process concentration value. Therefore, the user must enter a value adjusted by an amount proportional to the change in the concentration. For example, suppose the concentration is 3.80% when the sample is taken and 3.85% when the test result is entered. If the results of the analysis of the grab sample is 3.89%, the user should enter $(3.85/3.80)(3.89)$ or 3.94%.

LIMITATIONS

There are two important facts to keep in mind about calibration methods described above. First, the calibration is done at a single point. There is no certainty that the cell constant calculated at the calibration point will be exactly the same at other conductivity values. However, toroidal conductivity sensors are usually highly linear. Therefore, as long as the sensor is used in the recommend conductivity range, the cell constant will be reasonably close to the value at the calibration point. For example, when a Model 228 sensor is used with a Model 1056 analyzer between 0.015 and 1500 mS/cm, the expected variability in the cell constant is $\pm 1\%$. Second, the analyzer and sensor are calibrated as a unit. Thus, any error in the conductance (at the calibration point) measured by the analyzer is automatically incorporated into the cell constant. When the sensor is used with a different analyzer, which may have a slightly different error at the calibration point, the cell constant may be slightly in error.

ACCURACY

The accuracy of the calibration depends on the calibration method. For calibration against a standard solution, the accuracy depends on the accuracy of the standard itself and the accuracy of the temperature measurement. For routine work using a good quality standard and thermometer, the uncertainty is probably between 0.5 and 1.0%. For calibration against a referee instrument, either in-process or using a grab sample, the errors are greater. Error in the conductivity measured by the referee instrument, which includes the uncertainty in the conductivity of the standard used to calibrate it, and the variability of the cell constant over the operating range of the referee sensor are the major causes of the increased error. Of course, if the in process calibration is done at the same conductivity the referee instrument was calibrated, the error will be less. For calibration using a grab sample, the error will be even greater. The additional uncertainty arises from temperature measurement errors and failure of the temperature correction algorithm to perfectly correct for conductivity changes caused by temperature.



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