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Improving Robustness, Sensitivity and Simplicity of Potentiometric Sensors Through Symmetry and Conceptual Design

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Abstract: It is an enormous challenge to bring chemical sensing concepts from a controlled laboratory setting into the field while maintaining accuracy. In an environment of uncontrolled, fluctuating temperatures and a lack of repeated calibration, sensor reliability can rapidly deteriorate the accuracy. Today, many sensing concepts are explored for home use or as wearable sensors, and it is paramount to understand and optimize the chemistry for reliable measurements to become possible. This review focuses on the well-established class of potentiometric sensors, mostly known for the measurement of pH, with a range of electrolytes, and how conceptual advances can be used to make them as robust and sensitive as possible. While drawing from recent work of the group at the University of Geneva, the importance of symmetry is stressed to minimize the influence of temperature. The development of self-powered sensing systems that no longer require a battery is explained. This is then connected to protocols in which the sensitivity of these sensors can be reliably improved beyond that dictated by the Nernst equation.

Keywords: Chemical sensors · Constant current coulometry · Ion-selective membrane electrodes · Self-powered sensors

Eric Bakker has been the Chair of Analytical Chemistry at the University of Geneva since 2010. Born and educated in Switzerland, he studied Chemistry at ETH Zurich where he graduated with a doctoral degree in 1993 before moving to the United States for a postdoc and subsequent faculty appointments at Auburn University and Purdue University. He was promoted to Full Professor in 2003. In 2007 he moved to Curtin University in Perth, Australia as Director of the Nanochemistry Research Institute and was called to the University of Geneva three years later.

1. Motivation and Challenges for the Sensing of Chemical Parameters Away from the Controlled Laboratory

It is today imperative to monitor key chemical parameters with appropriate accuracy, as often as needed and at the appropriate location. But at the same time, there is a strong desire to democratize such tests so that they can be performed away from a controlled laboratory and even by people who have no or limited technical training.[1] From an analytical science perspective, this is an important challenge. The individual steps of sampling, aliquoting, sample preparation, injection, separation, detection, calibration and data treatment should be integrated into a single device and made invisible to the end user. Add the ability to perform continuous measurements and one can start realizing wearable sensors that operate autonomously for prolonged periods of time.[2]

At the heart of such integrated systems are chemical principles and materials that have been developed and optimized by analytical chemists for this very task. Key examples that come to mind are glucose test strips and continuous glucose monitors.[3] The former was a revolution in diabetes management since one single drop from a finger prick allows for a sufficiently accurate measurement of glucose for this application. These single tests are based on enzymatic reagents and integrated into an electrochemical test device that requires no individual calibration. While most of these tests work by amperometry as methodology, one example from Abbott uses capillary action to draw the sample into a confined channel of known volume to perform an exhaustive measurement by coulometry.[4] The electrochemical reaction is performed until all glucose has turned over, making the measurement independent of temperature and requiring extremely small sample volumes of the order of 300 nL.[5]

Continuous monitoring devices, on the other hand, are even more challenging to make since the device must be operationally stable for many weeks without any user intervention. Electrochemical glucose sensing principles have been adapted to work with a needle that penetrates the skin so that it can be placed into the interstitial fluid through a wearable device.[6] Another successful approach is by molecular recognition using polymeric diboronic acids, using fluorescence as the readout. The entire sensor is implanted under the skin to continuously sample the interstitial fluid. With such continuous monitoring devices the data are typically transmitted wirelessly for communication with a mobile phone.[3b,7]

With blood gases and electrolytes, the challenges are more important since the measuring range for most electrolytes is much narrower than that of glucose and single use test strips cannot be calibrated. Moreover, the readout principle is by potentiometry, not amperometry, giving a logarithmic dependence of the signal on ion activity.[8] If one tolerates an error of 5% in the concentration readout, which is already substantial, the uncertainty in the potential can be no more than just 1.3 mV. If the analyte is a divalent ion, such as calcium, the error can be no more than 0.6

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mV.[8] This is extremely challenging to achieve without a calibration step and careful temperature control. Unfortunately no single use test strips for blood electrolytes are currently available on the market. It would therefore be highly desirable to achieve a higher sensitivity for such ion sensors.

An additional challenge is the power consumption of electrochemical sensors. We are facing a paradox, since the awareness for environmental sustainability runs completely contrary to our demands for connectivity and digitalization, which requires battery powered devices. We need to take a step back and propose chemical and conceptual solutions to sensing that are self-powered and draw the power needed for the readout directly from the measurement itself. Self-powered amperometric sensors are already well explored,[9] but this is less obvious with potentiometric probes that are read out at zero current.

This review describes how some of these challenges are being overcome in our research group with conceptual advances with potentiometric sensors.

2. From Symmetry to Robustness

Potentiometric sensors need to exhibit adequate robustness if they are to be successfully used in real-world environments. This is especially important for applications in which environmental factors, such as temperature and pressure, vary in unpredictable ways. For many years now, solid contact ion-selective electrodes have been developed in view of making them more compatible with applications in the field.[10] Such electrodes are simple to make, are pressure insensitive, can be easily miniaturized and work in any desired orientation. This is in contrast with traditional electrode designs exhibiting an inner electrolyte solution.

Solid-contact membrane electrodes have one drawback that may have been overlooked in the past. The potential at the membrane–electrode interface and that at the reference element are not based on the same redox couple since reference electrodes are based on the Ag/AgCl/Cl^-(*aq*) couple, which is very different from the inner redox element of a potentiometric probe. The temperature-dependence of the two reduction potentials will therefore invariably result in a cell potential that changes with temperature.[11] This is in addition to the temperature-dependent electrode slope, the temperature-dependent ion activities and pH values of buffers.

Our group has aimed to minimize the effect of temperature on the cell potential by a duplication of elements in the measurement cell (see Fig. 1).[12] Two indicator electrodes of the same type are measured against each other, but one is immersed in a solution of known composition. A reference element placed in this solution is connected to the same reference element in the sample solution to form a closed bipolar electrode. The indicator electrode is now measured against a similar indicator electrode, canceling the temperature influence. In analogy, the reference electrode is now also measured against a similar reference electrode. Because the reference electrodes are connected by wire, the two compartments can be placed anywhere as long as temperature changes reach both electrodes at the same rate. Fig. 1 shows this arrangement for the measurement of pH with a solid contact polymer membrane electrode. The temperature influence is indeed dramatically improved compared to the classical non-symmetric configuration.

The concept of symmetry has also been used to improve the performance of potentiometric probes upon first solution contact, without any prior conditioning.[13] While proper conditioning in an electrolyte solution containing the analyte ion of interest gives the best potential stability, this is often not possible with routine measurements. In a single use device, for example, the measurement must be adequate upon initial solution contact. In a symmetrical arrangement a normally drifting potential reading can be greatly stabilized by measuring against a similarly drifting reference element: where the asymmetric membranes

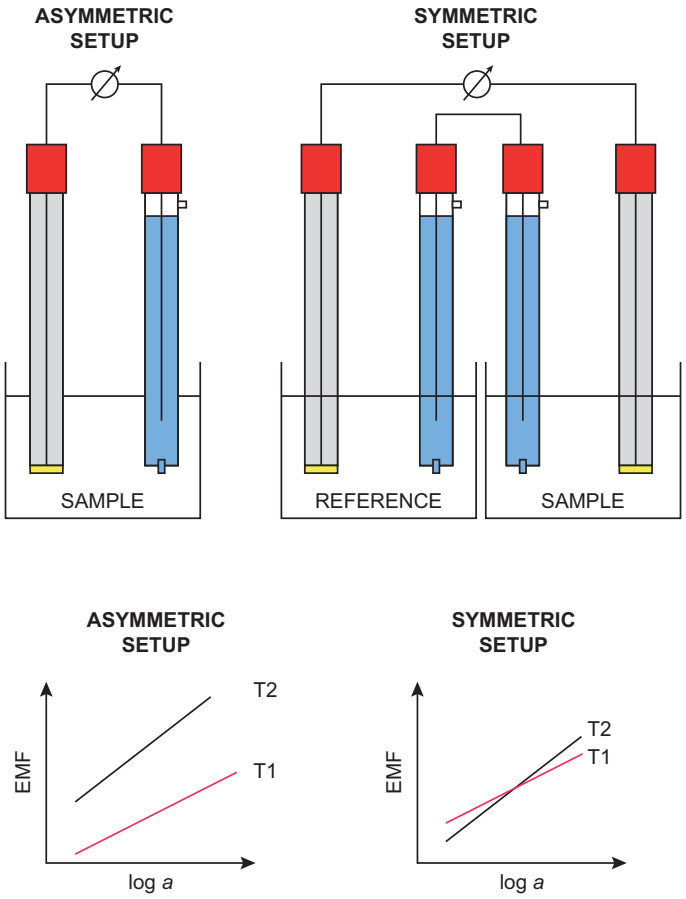

Fig. 1. Left: Solid contact potentiometric sensors tend to be measured against an Ag/AgCl-based reference electrode, which creates an asymmetry of the respective reduction potentials. As a result, the zero point of the potential lies outside the calibration curve and temperature changes not only alter the electrode slope but also the intercept in unpredictable ways (bottom left). Right: Asymmetric sensors can be made symmetric by duplicating each electrode as shown. The reduction potentials are made to cancel out and the composition of the left compartment can be adjusted to give a zero point in the middle of the calibration curve. This minimizes the influence of temperature on the calibration curve (bottom right).

took about half an hour to reach a potential drift better than 0.5 mV min^{-1}, the symmetrical arrangement reaches this value after about only 2 min.[13]

Moreover, the symmetric setup allows one to control the zero point of the cell to the middle of the calibration curve by adequately adjusting the composition of the reference electrolyte.[12] The zero point is the sample activity at which the cell potential is zero and the influence of temperature on the potential is smallest. An adjusted zero point greatly minimizes uncertainties originating from the temperature dependence on the electrode slope. A Nernstian slope of 59.2 mV suggests that a hypothetical potential readout of 400 mV gives a zero point that is different from the sample activity by 6.8 orders of magnitude. A variability of the electrode slope by just 1 mV results in an error of 6.8 mV. If the zero point is kept close to the sample activity one can greatly minimize this influence.

Is it always wise to use a traditional reference electrode with a concentrated electrolyte solution such as 3 M KCl? In some cases, in which the sample electrolyte composition is sufficiently constant, it may be advantageous to instead use a solution with a similar composition as the sample. Indeed, a freshwater sample spiked with about 150 µM $NaNO_3$ and in contact with the unmodified sample was calculated to give a liquid junction potential of just 0.3 mV, which seems attractive.[14] In such a case, the indi-

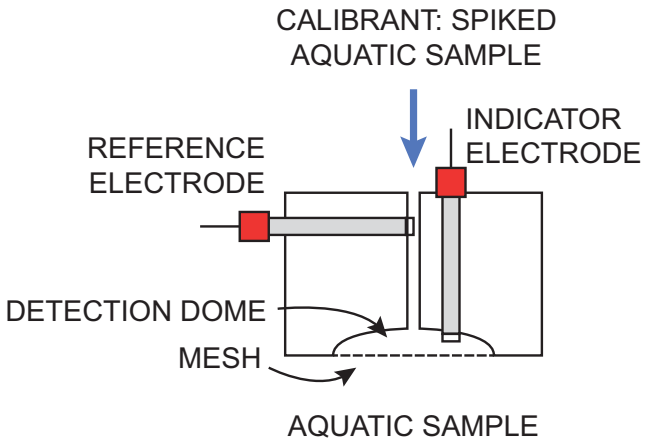

Fig. 2. Design principle of a submersible probe to detect ionic species (here, nitrate) in freshwater aquatic samples.[15] The reference electrode is of the same design as the indicator electrode and immersed in the same aquatic sample but spiked with a known concentration of analyte. This maintains the ionic strength between the two solutions, keeping the activity coefficients very similar and minimizing the liquid junction potential. Calibration is performed by displacing the aquatic sample from the shown sample dome. This design requires very little power since the pump is only actuated during calibration.

cator electrode in the sample solution can be directly measured against the indicator electrode in the sample solution using an open junction, without Ag/AgCl elements (see Fig. 2). If we can assume that the activity coefficients for the analyte ion are the same in both compartments it should be possible to calibrate for concentration instead of activity since the two activity coefficients cancel out in the equation for the overall cell potential.

This may give guidance on how one can achieve robust potentiometric probes for practical applications. If the electrolyte matrix is reasonably constant one may use a spiked sample as the reference compartment, in contact with a second indicator electrode. The symmetrical design minimizes temperature-dependent potential fluctuations. The liquid junction potential should remain small. The design also minimizes the zero point since the two solutions remain reasonably similar. Activity coefficients in the two compartments remain similar, allowing one to calibrate for concentrations.

These insights were recently implemented in the design of a submersible probe for the monitoring of nitrate in aquatic freshwater samples, see Fig. 2.[14] To minimize power consumption, the indicator electrode makes solution contact in a domed recess that is separated from the sample by a metal mesh separator. This design allows for the spontaneous convective equilibration between the dome and the environmental sample. To perform a one-point calibration, a pump is actuated to displace the sample from the recess with the reference solution. This probe was fabricated in house and successfully tested in the laboratory and in the field.[14]

3. Self-Powered Potentiometric Sensors

The potentiometric probes used today in clinical analysis are mostly based on hydrophobic polymeric membranes that are doped with appropriate sensing components. They contain a lipophilic ion-exchanger and a so-called ionophore, a lipophilic complexing agent that selectively recognizes the ion of interest. Traditionally, such membranes are interrogated against a reference electrode by a high impedance input voltage meter that contains an operational amplifier that functions as voltage follower. In this configuration, no appreciable current flows through the cell. Without current, however, no work can be performed, and such devices require a dedicated power source. Additionally, it is well established that potentiometry is a zero current method. A residual current flow would require electrochemical turnover reactions at

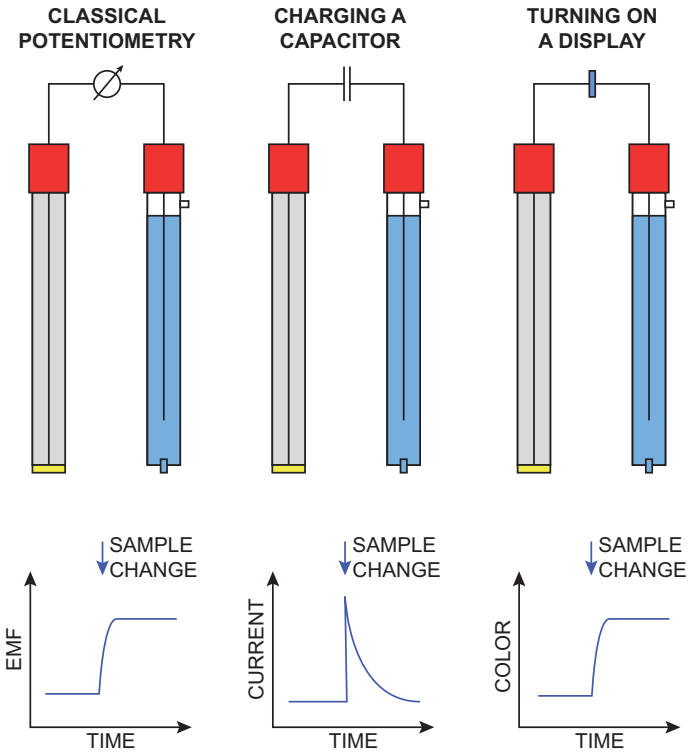

Fig. 3. Left: In classical potentiometry a high impedance input voltmeter is placed between the indicator electrode (left electrode) and the reference electrode, giving a potential change upon changing the sample composition (bottom). Middle: If the voltmeter is replaced by a capacitive element, the short circuiting results in a transient current (bottom), charging the capacitor so that the circuit can be used to do work.[16] Right: Placing an electrochromic element instead of the capacitor between the electrodes gives a direct, reversible self-powered display of the potentiometric response.[17] A color change is observed in analogy to a potentiometric response (bottom). The vertical arrow indicates the time at which the sample composition is changed to trigger the signal.

the electrode, which may be influenced by charge and ion transfer kinetics. This would be undesired. So how can we possibly perform work from such sensors?

One should remember that the zero current condition should be reasonably valid at the time of measurement. This means that a transient current may flow before the measurement is being made. This hypothesis was successfully demonstrated by directly connecting a capacitive element between the sensing and reference electrodes.[15] In such a short circuited cell, the potential developing between the two electrodes upon solution contact will result in current flow until the cell potential reaches an overall potential of zero. Current flow, however, will start charging the capacitive element and stop when the voltage over the capacitor cancels the voltage difference between the two electrodes. Consequently, the capacitor can be disconnected from the cell, measured separately with a multimeter, or otherwise used to perform work (see Fig. 3 middle).

Of course, one can now use additional electronic components to expand the functionality of the resulting device. In one example, a diode was placed in series with the capacitive element to keep the capacitor from discharging again.[17] In this way, one may construct a sensing device that has memory, even if the sample composition changes back to the basal level after a perturbation. A recent expansion of this concept made use of three capacitive elements, one of which contains no diode elements.[18] The comparison of the potentials over the different capacitors makes it possible to identify whether concentration excursions have occurred in either a positive or negative direction before the measurement was made.

These findings made it possible to integrate an electrochromic element instead of the capacitor described previously (see Fig. 3 right). The function is fundamentally similar, but the element indicates the voltage change by an optical response. The first example with a short-circuited design used an electropolymerized Prussian Blue film in contact with an appropriate electrolyte solution and electrically connected to the potentiometric probe.[16] The circuit was closed by connecting the reference electrode in the sample with the Ag/AgCl element in the solution of the electrochromic element. This forms a so-called closed bipolar electrode arrangement, meaning that here the electrical connection to the Prussian Blue–Potentiometric Sensor pair is through solution contact, not by wiring. In principle, therefore, any number of bipolar electrodes can be placed in such a circuit to all work at the same time. The electrochromic display works reversibly, with the absorbance originating from the Prussian Blue/Prussian White electrochemical conversion following the potential for the sensing circuit in real time. This is an optical sensor in which the color generation is physically removed from the sample, which is advantageous since opaque or turbid samples can also be measured. It is fully self-powered since the display spontaneously follows the sensor output. Of course, one must adjust the working potential range of both the display and the sensing circuit, which may be accomplished by chemical composition adjustments.

To make further progress our group explored alternative display materials that could be used with this principle. In an attractive example, electronic paper (e-paper), commonly used in electronic books, was found to be an excellent electrochromic material.[19] The darkening of the pixel depended reversibly on the applied potential and remained indifferent with a constant applied voltage. Commercially available e-paper arrays with appropriate electrical connections can be easily connected to a series of potentiometric probes to achieve simultaneous detection of multiple analyte ions without an external power source.

Similar to most optical sensors, including colorimetric pH paper, the sensitivity of an optical readout remains quite limited. A visual readout already makes it challenging to discern concentration changes of the order of factor 2 or so, which limits the practical usefulness of this principle. To overcome this, an intermediate charging step was proposed in which the sensing electrodes were not connected to the display pixel but to three parallel capacitors.[20] In this way the capacitors are all charged to the same value. Using a homemade slider, the three capacitors are then connected serially to the display pixel. The serial connection now sums the voltages and results in an approximately 3-fold sensitivity. In principle, of course, any reasonable number of capacitors could be used to increase the sensitivity even further. This shows the power of combining electrochemical and electronic principles and components to achieve functionalities that have not been possible before.

An alternative approach to achieving a better readability of the signal is to design a distance-based approach using an array of display pixels that are all connected in parallel with the sensor circuit. For this to work, each consecutive pixel must indicate an incrementally decreasing potential, which is achieved by connecting increasing capacitances in series with each pixel. The principle was successfully demonstrated with an e-paper pixel array [21] but for it to work best, the dynamic range for each pixel should be as small as possible so that a transition from dark to bright can be easily observed at a pixel location that changes with sample concentration.

4. Increasing the Sensitivity of Potentiometric Sensors

The Nernst equation is the fundamental basis to describe the operation of potentiometric sensors, which states that the cell potential is proportional to the logarithmic ion activity with a sensitivity (slope) of 59.2 mV/z where z is the ion valency. Small potential uncertainties give large measurement errors, as stated in the introduction above. A few years ago a novel approach emerged in the literature, coined *constant potential coulometry*.[22] It is essentially related to the concepts discussed in the previous section in which a capacitive element is placed between the indicator and reference electrode. Two changes are made here, however. The cell potential is not zero but held to a constant value by a potentiostat. Further, the transient current over the capacitive element is recorded and serves as signal after integration with time to give the charge (see Fig. 4 middle).

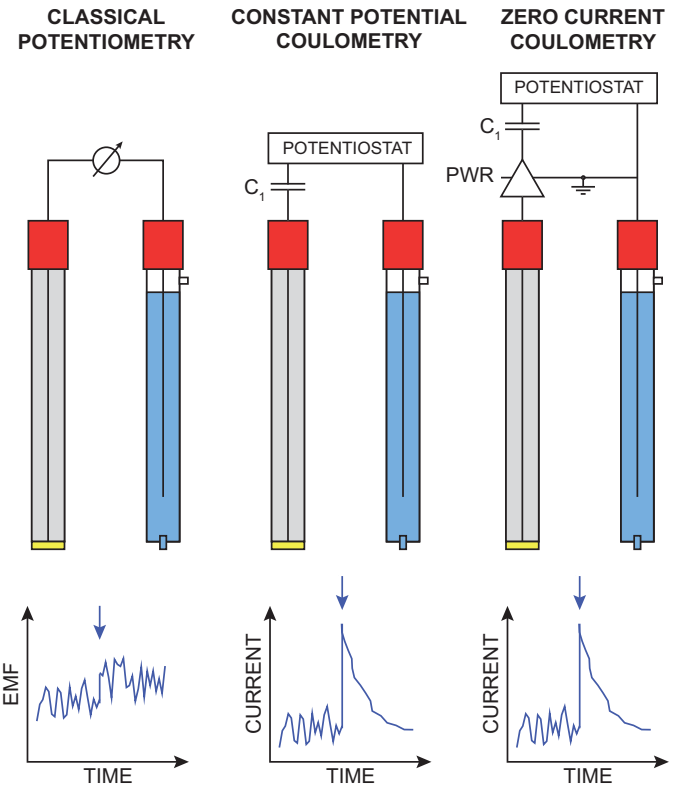

Fig. 4. Left: In classical potentiometry, small concentration changes in the sample result in a subtle change of the baseline potential, which is difficult to distinguish from drift (bottom). Middle: The sensitivity can be improved by allowing for a transient current over a capacitor (bottom), which is driven by the mismatch between the voltage dictated by the potentiostat and the open circuit voltage over the measurement circuit.[24] However, the current flows through the circuit, resulting in possible signal bias. Right: The use of a voltage follower allows one to pass the current independent of the measurement circuit.[25] While the output is the same as in constant potential coulometry (bottom), any electrode of choice can now be used, including highly resistive pH glass electrodes.

The reason why this method can give higher sensitivity than potentiometry lies in the generation of a signal peak upon changing the sample from a reference solution to the sample of interest. In potentiometry such a change would simply result in a potential step (Fig. 4 left). As known from chromatography and spectroscopy, a peak is much easier to isolate and baseline correct than a change of the baseline, which is more easily buried in background noise and signal drift when the change becomes small.

Our early work used an electronic capacitive element and a switch valve in a fluidic setup to abruptly switch from the reference solution to the sample and vice versa.[23] Indeed, extremely high precision down to about 60 µpH was found for 1 mpH step changes, corresponding to 4 µV or 0.01% acidity change. This may be unprecedented for a potentiometric probe. Indeed, the

same experiment observed by potentiometry did not give meaningful results.

Further work aimed to replace the repetitive fluid handling steps with an instrumental control that could trigger the current transient at a precise time.[24] The idea was to measure the potential of the reference sample, store the value in memory and apply this potential at the desired time after the sample is introduced into the cell. This should allow for repetitive pulsing without having to change the sample, making the method as simple as possible. An appropriate instrument was developed and successfully demonstrated. It was found, however, that in this mode the potential drifts originating from the current transients, which impose concentration polarizations in the membrane, have a direct bearing on signal drift.[24] When aiming to expand the method to other membrane materials, such as the well-established fluoride single crystal electrodes, the approach also failed.[25] This was overcome by treating the indicator electrode as the reference electrode in the measurement circuit, allowing for the transient current to flow through the reference element instead. This greatly reduced signal drift. After some optimization a reproducibility of just 0.02% was found for the measurement of fluoride using this method.[25]

High impedance sensing elements such as the pH glass electrode or resistive microelectrodes cannot be measured by connecting them directly to ground. We have aimed to overcome this last limitation by implementing a voltage follower in the circuit (Fig. 4 right).[26] The transient current now no longer flows through the measurement cell and essentially any practical electrode can be measured by constant potential coulometry. The approach was successfully demonstrated with an oceanographic pH electrode, giving a precision of 64 μpH with pH increments of 0.01 units.[26,27] The measurement is now indeed as simple as that for a classical pH electrode.

It should be emphasized that any of the electronic approaches described above separate the measurement from the calibration step in time to gain practicality and convenience. This means that potential drifts may now more easily become the limiting factor in achieving high sensitivity and precision. To avoid frequent recalibration, one should work with sensors of appropriate potential stability.

5. Conclusions

While chemical advancements to find improved materials for potentiometric sensors have been the main focus in this research field, conceptual considerations should accompany that important effort. A chemically stable solid contact transducing material is important for signal stability, but the measurement cell should be symmetrical with regards to that transducing element. The zero point of the measurement cell should be optimized to lie in the middle of the measuring range to minimize temperature influences. Reference electrode configurations and electrolytes should correspond to these requirements and be adapted to the application. Self-powered sensing systems on the basis of potentiometry are realizable, of course within limits. Allowing for a transient current flow gives scope to integrate electronic circuit elements in potentiometric measurement cells to create sensors that have a memory function. Importantly, it allows one also to overcome the Nernst limit so that the sensitivity may be improved while maintaining a fundamental understanding of these important devices.

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