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Application Note

In this application note we explain how to use the IR-compensation module in PalmSens potentiostats. We start explaining the terminology, the issues of ohmic drop in an electrochemical cell and how the IR-compensation can help. Finally, we show the main methods to determine the uncompensated resistance.

1 What is IR-drop and uncompensated Resistance?

IR-compensation or *ohmic drop compensation* is a compensation of the residual resistance between RE and WE (or S). While this resistance is negligible for most electrochemical cells, it can be significant for high currents and poor-conductive electrolytes.
When using a potentiostat we may think that the applied potential is only applied across the working electrode's solid-liquid interface. However, in a real system, the potentiostat applies the potential between the reference electrode (RE) and the working electrode (WE). Between the WE and RE interfaces there is some electrolyte, which acts as an Ohmic resistor. This resistance leads according to Ohm's law to a potential drop of current times resistance or I*R (see Figure 1).

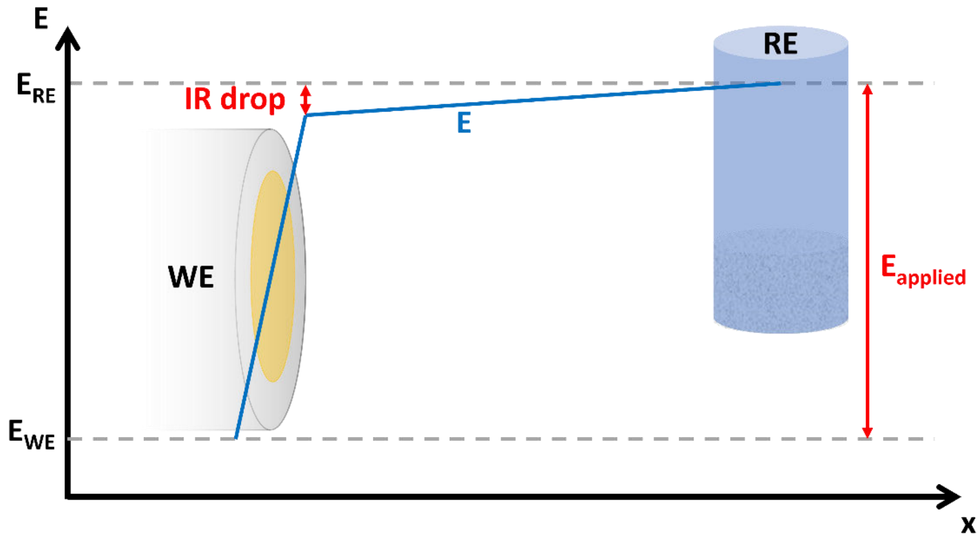

Figure 1 Scheme of a potential profile between RE and WE with highlighted IR drop

For most experiments the IR drop is negligible due to low currents or the low resistance R_u between the WE and RE. For example, an R_u of 10 Ohm and a current of 100 µA leads to an ohmic drop of (10 Ohm * 100 µA =) 1 mV. A potential shift of 1 mV is not relevant for most experiments. Experiments with higher currents or solutions with low support electrolyte concentration and thus low conductivity can have significant IR drops, which need to be compensated to achieve accurate results.

2 IR Drop Compensation Module

For compensating the IR drop special hardware is required that can correct the applied potential dynamically during the measurement. The module provides positive feedback to compensate for the IR drop between the reference electrode and the outside of the double layer of the electrochemical cell.

2.1 Compatible Devices

IR Compensation is available for the following models of PalmSens potentiostats:

- PalmSens4: available as an optional *in-factory* add-on module. It can be added at a later stage, but it requires the device to be returned to the factory for installation

- EmStat4X: included as standard feature for both LR and HR versions
- Nexus: included as standard feature

2.2 Positive Feedback

All PalmSens IR Compensation modules work by means of positive feedback. In a positive feedback loop an entered stimulus will produce a result that increases the stimulus. In this case the feedback loop dynamically adds more potential to the entered potential to compensate for the IR drop.
This is achieved with a 16-bit multiplying digital-to-analog converter (MDAC) in the module which scales the output of the current follower operational amplifier to provide a positive feedback voltage which is proportional to the current through the cell. The compensation voltage is added as a summing point before the control amplifier and thus increases (or decreases if the current is negative) the applied potential to counteract the IR drop.

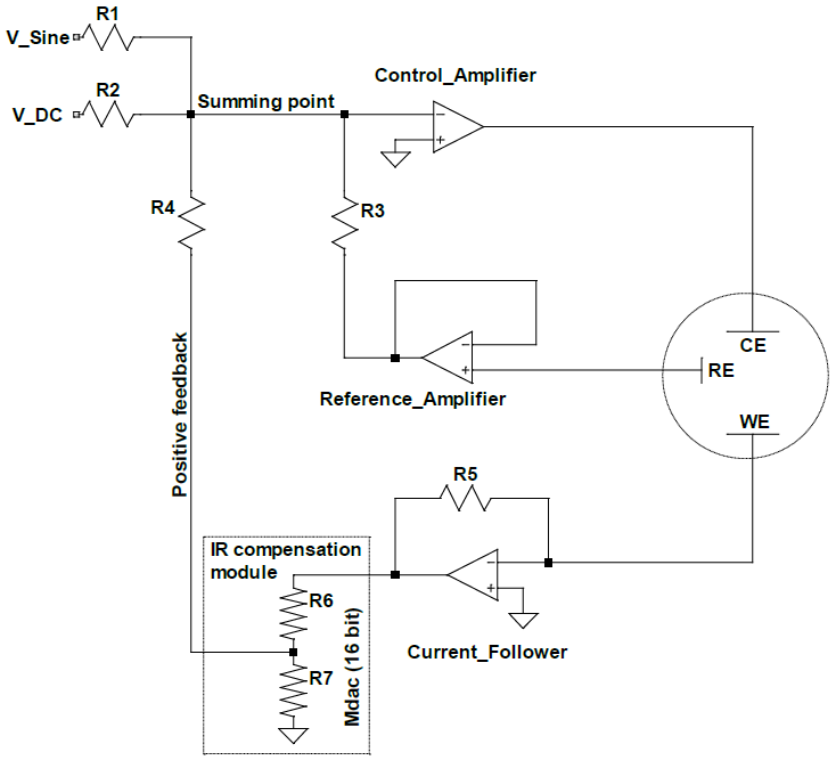

Figure 2 Scheme of the IR compensation module's circuit in a potentiostat

Positive feedback allows for fast scan rates up to 10 V/s, depending on the characteristics of the cell. If the potential error to compensate for becomes close to the value set for $E_{applied}$, the system might become unstable. Using IR compensation limits the measurement bandwidth to 10 kHz.
As mentioned, the potential is increased proportionally to the measured current. The proportionality factor is the uncompensated resistance R_u. This compensation method uses a single R_u value during the whole measurement. This implies an assumption of a constant R_u during all the experiment. This assumption is reasonable for most electrochemical cells as the electrolyte resistance between WE and RE interface is almost constant. Thus, the value of R_u must be entered manually into the software, which means that the R_u must be determined before the measurement. In Chapter 4 we discuss the main methods for determining the R_u.

2.3 Current Interrupt Modules

The Current Interrupt is simple method for IR compensation, used mainly in corrosion studies. This method is not used by PalmSens. It is not common to describe what you do not have, but due to the widespread use of this method we would like to clarify that it is not the method used by our potentiostats.

The current interrupt method performs an additional small measurement between two points of a measurement. The current is interrupted for a short moment. Consider the Randles circuit (see Figure 5) as representing the cell, so we have a parallel resistor and capacitor (RC) and a series resistor R. The latter is the R_u. When the current is interrupted, the potential across R_u immediately drops to 0, while the capacitor in the RC slowly decays. The drop of the potential is measured, and R_u calculated. These processes are very fast and usually require hardware with fast data sampling.

The main disadvantage is the interruption itself. While it is brief, it might interfere with most electrochemical experiments. It has been used in corrosion studies due to the nature of slow changes for most corrosion cells.

3 How to operate the IR compensation

When a potentiostat with IR drop compensation module is connected, you can find in the Method Editor of PSTrace a field to enter the resistance you want to compensate for after pressing the …-button. The Method Editor is the field on the left side of the PSTrace window, where all the measurement parameters are set.

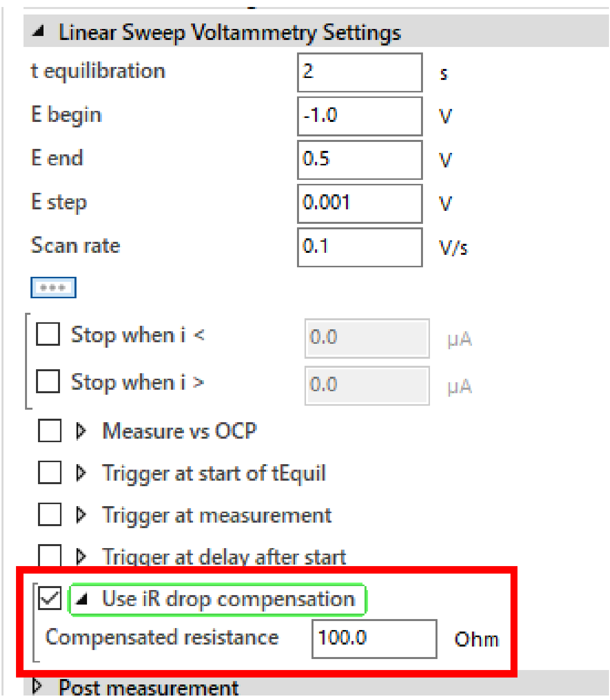

Figure 3 Activate IR drop compensation and define the resistance to compensate in PSTrace

Depending on the entered resistance, some current ranges might not be available. If this is the case, you will get an error message from PSTrace:

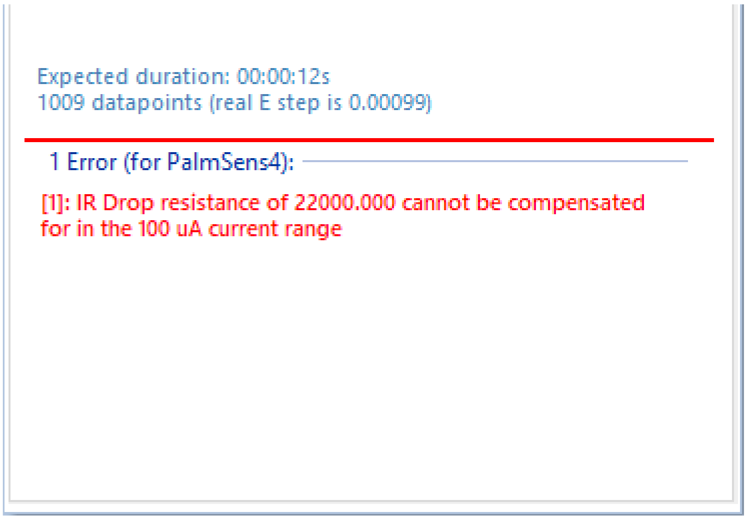

Figure 4 Error in PSTrace when a selected current range is not suitable for the entered resistance to compensate

There is a limit of resistance that can be compensated for each current range, as the higher the R_u is, the higher is the potential to be summed. Adding a high value of potential could lead to instability, overload and exceed the device capability. Thus, the error messages are intended to avoid these issues.

4 Determining the Uncompensated Resistance R_u

Knowing the uncompensated resistance R_u of your cell allows you to use the IR compensation module to reduce the IR drop to a negligible level. There are multiple ways to determine the uncompensated resistance R_u in your cell. Most of these techniques use the Randles circuit as a basis for their approach. We are looking at very fast processes, so the Warburg element can be ignored. Thus, we consider the simplified Randles circuit with the double layer capacitance C_{dl}, the charge transfer resistance R_{ct} and the electrolyte resistance, which is the same as uncompensated resistance R_u.

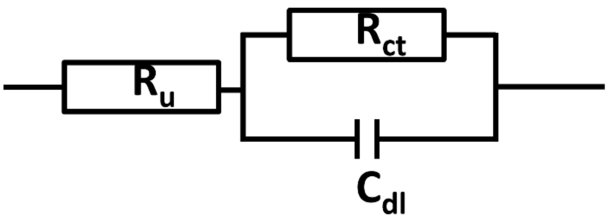

Figure 5 Randles Circuit

4.1 Electrochemical Impedance Spectroscopy

Most likely the quickest and most convenient way to determine the uncompensated resistance is impedance spectroscopy. Electrochemical Impedance Spectroscopy (EIS) is an *Alternate Current* (ac) technique, i.e. the applied and measured potentials or currents are sine waves.
EIS is a complex topic, and we already have multiple articles and videos about this topic and if you would like to dive deeper into this technique, we recommend the following articles:
https://www.palmsens.com/knowledgebase-article/electrochemical-impedance-spectroscopy-eis/

https://www.palmsens.com/knowledgebase-article/impedance-analyzer/
https://www.palmsens.com/knowledgebase-article/bode-and-nyquist-plot/
https://www.palmsens.com/knowledgebase-article/pstrace-tutorial-14-eis-parameters/

When an ac voltage is applied across the circuit in Figure 5, the impedance that depends on the frequency of the ac voltage. We are only interested in the R_u, which is frequency independent. The other resistor R_{ct} has a frequency-independent impedance too, but the capacitor C_{dl}'s impedance depends on the frequency. The higher the frequency, the lower the impedance of C_{dl}. At sufficiently high frequencies all current will flow through R_u and C_{dl}, but C_{dl} contribution is negligible, so you only see R_u in the spectrum.
As higher frequencies may have stray capacitance effect (i.e., unwanted capacitance from cables or other metal surfaces), it is advisable to measure a full spectrum and acquire the R_u by equivalent circuit fitting. In the following video tutorial we explain how to perform equivalent circuit fitting:
https://www.palmsens.com/knowledgebase-article/pstrace-tutorial-10-equivalent-circuit-fitting/

The R_u is independent of the parameter *E dc* that is applied during the EIS, this means you can sample it at multiple values, but keep in mind that one requirement for an EIS measurement is a steady state.

4.2 Oscillation due to overcompensation

If your potentiostat is not very fast and is not EIS-capable, a simple way of determining the right uncompensated resistance R_u is checking at which resistance you are overcompensating.
Using positive feedback can lead to instabilities in your systems. When you are compensating with more resistance than your R_u the compensation will trigger the feedback loop of your potentiostat, and you will observe overshoots and ringing. This means the potential will rise beyond the set value and then will go back to the set value. In the case of ringing there is a small decaying sine wave. These are harbingers of oscillation, a state where the feedback loop of the potentiostat is permanently overcompensating, leading to a signal that looks like noise.
You can exploit this effect by performing a series of measurements with increasing compensated resistances. When the system starts to oscillate the compensated resistance is too high. A ringing effect is still acceptable. Choosing around 90 % of the value where your potentiostat starts to show strong ringing or oscillation is usually close enough to R_u without triggering the oscillation.

4.2.1 How to perform this

The idea is to perform fast potential steps and observe how the potential reacts to this. These steps are not our real experiment, we just use them to determine R_u, so small potential steps are preferred to keep the impact on the system low. The potential before and after the step should not trigger Faraday currents.

1. Connect your cell and the PalmSens4 as usual.
2. Choose *MultiStep Amperometry* as Technique.
3. Set a current range according to the expected current. We want to perform a fast measurement, so only a single current range can be active to prevent *auto-ranging*.
4. Set a *t equilibration* of 5 s.
5. Set a *t interval* of 0.0004 s, which is the lowest limit for the t interval.
6. Set the *Cycles* to 1 and the *Levels* to 3, because we want to make one steop forward and one step backwards. No repetition is required.
7. Set the *E level 1* to the potential before the step. And *t 1* to 0.02 s. We can keep each level short, to make the measurement fast.
8. Set *E level 2* to 50 mV higher or lower than *E level 1*. We want a potential step big enough to see a clear step, but small to keep changes to our system small. The duration *t 2* is also 0.02 s.
9. *E level 3* and *t 3* are the same as level 1.
10. Check in the segment *Record additional data* the box *Record WE potential*.
11. You can first perform a measurement with no IR-compensation.
12. Then perform multiple measurements with increasing resistances to compensate.

13. Look at the plots of E vs t. The R_u should be close to the resistance where the ringing begins. Just choose a resistance slightly beneath it (90 %).

It is helpful if you know roughly the R_u. If you don't know R_u approximately, choose a wider range for your compensation and choose big increments for the resistance to compensate. For example, perform measurements from 0 to 800 Ohm with 100 Ohm steps. Should you see an oscillation at 800 Ohm, perform measurements with smaller steps between 600 and 800 Ohm to see where ringing begins.

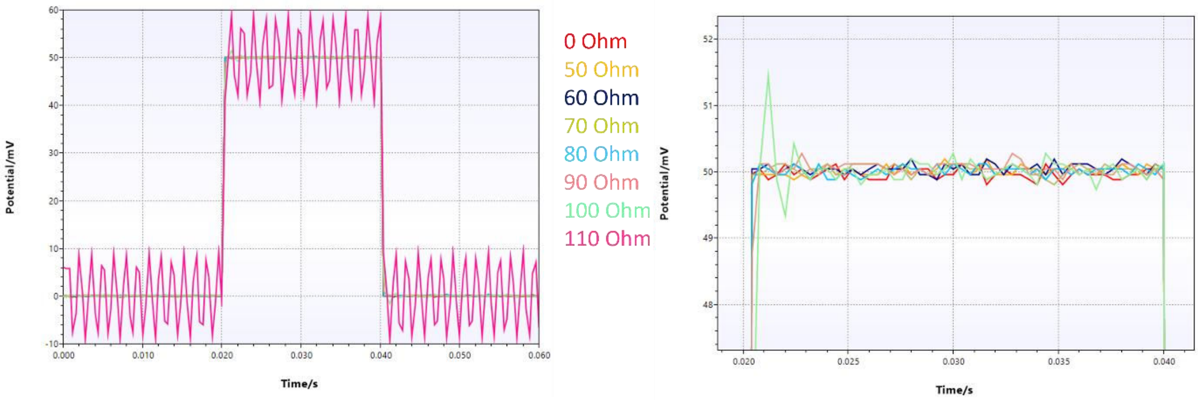

Figure 6 Measured potential of the WE during a MultiStep Amperometry with varying compensated resistances (left) and a zoom in (right)

4.3 Interrupted Current

A capacitor reacts slowly to changes of potential or current compared to a resistor. As explained in chapter 4.1, very fast changes of potential or current lead to a negligible impedance of the capacitor.

This is exploited by the interrupted current method. A constant current is applied to the system. Then the circuit is opened, which interrupts the current flow, leading to zero current. The uncompensated resistance will immediately react, and the potential drop across it is zero.

After that, the capacitor, or electrochemical double layer respectively, will discharge. A capacitor in parallel to a resistor, here the charge-transfer resistance, discharges exponential according to the equation:

$$E = E_0 e^{-\frac{t}{R_{ct}C_{dl}}}$$

Where E_0 is the initial potential and E the potential over time t. This means we need to measure the potential before the current is interrupted and right after the interruption that will be E_0 for the capacitor.

For this you need a potentiostat that can measure the very fast potential drop accurately. With that potential drop you can calculate R_u. Just divide the potential drop by the current value before the interruption. The time constant τ defines how fast this measurement must be:

$$\tau = R_{ct}C_{dl}$$

τ is the time in which around 63 % of the initial current is gone. For a successful measurement you need to measure at least 100 times faster.

4.4 Potential Step

Analog to the current interrupt described in chapter 4.3, you can also use a potential step to determine the uncompensated resistance R_u. For this technique, Faraday currents need to be avoided. A potential step is applied between two potentials and both potentials should not lead to a Faraday current. This means that the R_{ct} can be removed from the Randle circuit (Figure 5). This leads to an even simpler circuit as shown in Figure 7.

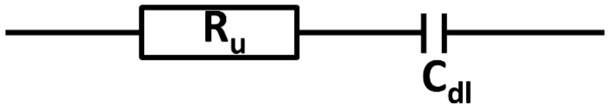

Figure 7 Circuit model for potential step

The potential step will change the amount of charge that the capacitor can store, and we are going to observe this change. For example, you could jump from 0.02 V to 0.03 V and observe the current flowing. From electronics it is known that the current during a charging or discharging decays exponentially according to:

$$I = I_0 e^{-\frac{t}{R_u C_{dl}}}$$

Here I_0 is the initial current caused by the potential step. Please note that here the time constant τ is in this situation defined by R_u:

$$\tau = R_u C_{dl}$$

An instrument with fast data sampling and precise timing is required to measure the current. Ideally, you could use the initial current step I_0 of the curve to calculate with the potential step ΔE the resistance R_u using Ohm's law, because during the potential step the capacitors impedance is negligible. This would require a very fast measurement but could be sufficient for a rough estimation.
A more accurate way is linearizing the curve, make a linear regression and use the intercept to determine I_0. The linear form of the equation for I is:

$$\log I = \log I_0 - \frac{t}{R_u C_{dl}} \log e$$

4.4.1 How to perform this

When you want to perform this technique, you need to consider multiple parameters. You need to avoid a Faraday reaction current, otherwise you have an additional current pathway across R_{ct} (see Figure 5). A low time constant τ will lead to inaccuracies because the charging is happening too fast. You need software that allows you to perform linear regression.

Considering the amount of work and possible sources for inaccuracies, EIS (see chapter 4.1) and overcompensation (see chapter 4.2) are both preferrable for uncompensated resistance determination.

1. Connect your cell and the PalmSens4 as usual.
2. Choose *Fast Amperometry* as Technique.
3. Set a current range according to the expected current. We want to perform a fast measurement, so only a single current range can be active to prevent autoranging.
4. Set a *t equilibration* of 10 s or long enough to have a stable current.
5. Set *E equilibration* to the potential before the step and *E dc* to the potential after the step.

6. A *t run* of 0.001 s should be sufficient.
7. Set a *t interval* of 0.00002 s, which is the lowest limit for the *t interval*.
8. Perform the measurement.
9. Export the data to the software where you want to perform the processing.
10. PalmSens4 has under the given settings a small delay before starting the recording. To compensate for that add 0.00016 s to the time. Plot the logarithm of the current lg I versus time t.
11. Perform a linear regression on the linear part of the curve to acquire the intercept.
12. The intercept is lgI_0. Convert it to I_0 and calculate Ru with $\Delta E / I_0$.