Temperature calibration of a potentiostat board with an ITS

I – INTRODUCTION

Intermediate Temperature System (ITS) is a compact temperature control unit based on the Peltier effect, used for the electrical characterization of materials. It is controlled by MT-Lab® software via an USB connection when used with the MTZ-35 impedance analyzer. The ITS is also controlled by a BioLogic potentiostat through its analog output using EC-Lab® software.

The ITS is temperature calibrated in the factory with the two provided PT1000 probes. For use with the MTZ-35 impedance, analyzer no calibration is needed. However, for a use with a potentiostat, calibration may be needed because of a drift or an offset on the Analog out/Analog In of the user's potentiostat board.

This technical note details the procedure to follow for the calibration of the Analog out, the Analog In1 and the Analog In2 of a potentiostat board used for the temperature control and measurement of ITS.

II– PRINCIPLE OF THE ITS CONTROL WITH A POTENTIOSTAT

The temperature of the ITS is controllable by a Potentiostat through the Analog out. Two temperature variables are measured: the ITS temperature termed here as "T_{base}" and the cell temperature labelled as "T_{cell}". These two temperatures are acquired as two external voltages through the two Analog inputs of the potentiostat board. Figure 1 shows the "T_{base}" and the "T_{cell}" Temperature measured by the two PT-1000 provided with the ITS.

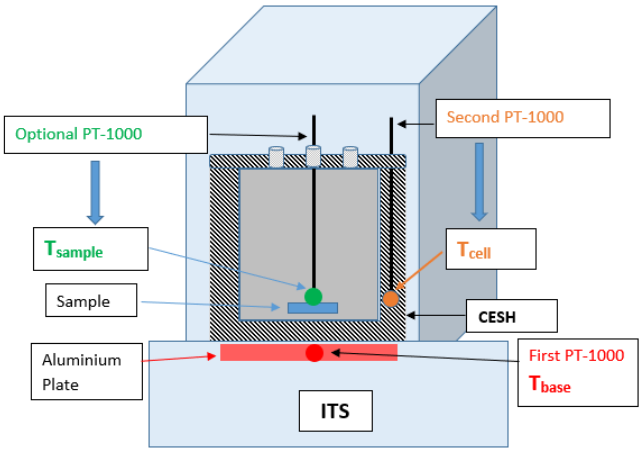

Figure 1: Schematic showing the location of the PT-1000 probes and their corresponding temperatures.

Note:

By default, two PT1000 are provided with the ITS. The first PT1000 is integrated into the ITS for measuring T_{base}. The second PT1000 is installed in the T_{cell} hole of CESH for measuring T_{cell}. An optional third PT1000 can be used with the ITS for measuring T_{sample}.

T_{sample} can also be measured by EC-lab (Instead of T_{cell}).

The calibration is carried out in two independent steps: a fast calibration of the Analog output and a second calibration of the analog inputs.

III– CALIBRATION OF THE ANALOG OUTPUT

The default calibration parameters of the Analog out of the ITS are given in EC-Lab® software. They can be viewed by clicking on "**External Devices"** tab and selecting TCU in "**Device Type"** box and ITS in "**Device Name**" box (Fig. 2).

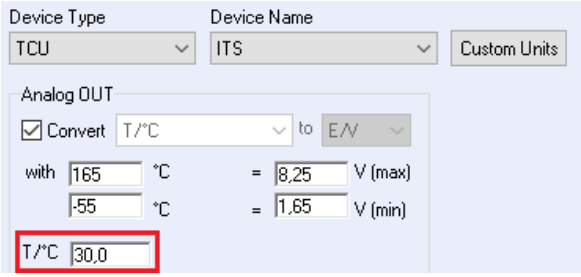

Figure 2: Default settings of analog out for ITS.

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The calibration of the Analog out consists of plotting the graph "setpoint temperature" against a series of voltage values". It is a linear relation between the temperature *T* and the voltage *E*:

$$T = a\,E + b \quad \text{where}$$

T is the output signal (Temperature on ITS).
a is the slope of the linear plot.
E is the input signal (Voltage set in EC-Lab®).
b is the offset temperature.

The calibration procedure of the Analog out is as follows:

- Connect the ITS to the potentiostat using the provided DB9-DB9 cable (Fig. 3).

Figure 3: Connection of the potentiostat board to an ITS using the DB9-DB9 cable.

- Open EC-Lab® software and connect it to the potentiostat.
- Load "**OCV"** technique (Fig. 4).
- Go to "**External Devices**" tab and select "Other" in "**Device Type**" and "Other" in "**Device Name**" box (Fig. 4).
- In Analog OUT section, select E/V variable in the "**Convert**" box and check it. Then set the values 10 V and 0 V as shown below.

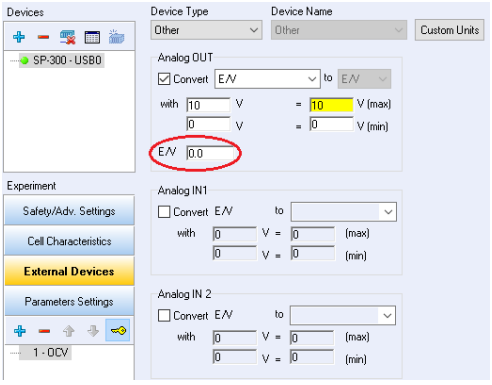

Figure 4: Analog Out settings for calibration with ITS.

- Set a first setpoint voltage (for example 3.0 V) in the circled box on the Fig. 4 and press on the yellow key button to validate the setpoint voltage.
- Read and note the temperature setpoint displayed on the ITS screen.
- Set another setpoint voltage value and read the corresponding setpoint temperature displayed on the ITS screen.

The table I summarizes the voltage values and the corresponding setpoint temperatures obtained for the calibration of the Analog out.

Table I: Summary of the setpoint voltages set in the Analog OUT section and the corresponding setpoint temperatures displayed on the ITS screen.

E/V	ITS setpoint temperature/°C
3.0	- 9.9
4.0	23.4
5.0	56.8
6.0	90.1
7.0	123.4

The points (*E*, *T*) are then used for plotting *E* vs. *T* linear graph (Fig. 5). The obtained linear relation is: $E = 0.03\,T + 3.297$ (Fig. 5).

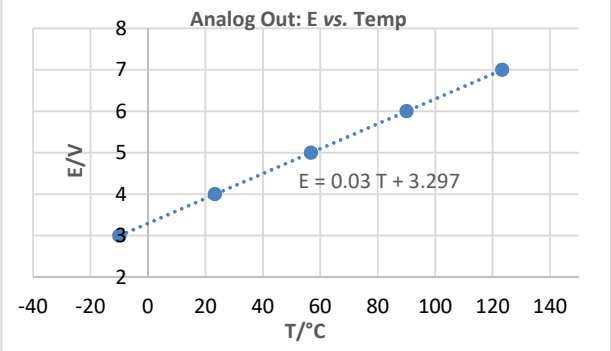

Figure 5: Voltage *vs.* Temperature plot of Analog Out.

Once the linear relation has been determined, two points (T_1, E_1) and (T_2, E_2) have to be set in the EC-Lab® software. By default, the voltage at - 55 °C and 165 °C are usually used in EC-Lab® for the voltage-temperature conversion:
For T_1= - 55 °C; E_1 = 1.647 V

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For T_2= 165 °C; E_2 = 8.247 V
The values obtained here are the same as the default settings given in EC-Lab® (Fig. 2).
The setting with these two points has to be checked by using at least two temperatures.

Note:

The low and high temperature values set in Analog OUT section have to be lower than - 40°C and higher than 150 °C respectively, as they are considered to be temperature limit values by EC-Lab® software.
A warm-up time of 30 mini is recommended before proceeding to the calibration. At this point, the calibration of Analog out can be performed quickly. It lasts less than 2 min.

IV– CALIBRATION OF THE ANALOG INPUTS

The T_{base} and the T_{cell} are acquired by Analog In1 and Analog In2 respectively. Analog In1 and Analog In2 operate in the voltage range ± 10 V. The user needs at least two setpoint temperatures T_1 and T_2 to calibrate each Analog input.

III-1 Step 1:

The first step consists of creating two variables: E_1 voltage for Analog In1 and E_2 voltage for Analog In2. The user should proceed as follows:

- On EC-Lab® software, select "**External Devices**" tab.
- Click on the "**Custom Units**" button (Top right on Fig. 6). The window in Fig. 6 appears. Click on "**Add**" button.
- Type "E_1" in the box "**Name**" and "V" in the box "**Unit**" (Fig. 6 left). Do the same thing for the variable "E_2" (Fig. 6 right)

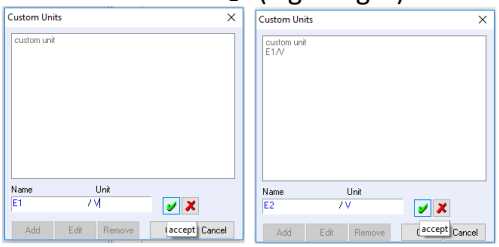

Figure 6: Addition of E_1 and E_2 voltage variables to the "External Devices" Tab.

III-2 Step 2:

Once the E_1 and E_2 variables have been created, the EC-Lab user can start the calibration of the Analog In1 and Analog In2 by proceeding as follows:

- The potentiostat has to be connected to ITS as shown in Fig. 3.
- Load Temperature control (TC) protocol on EC-Lab®.
- In "**External Devices**" tab, select TCU in "**Device Type"** box and ITS in "**Device Name**" box.
- Replace the T/°C by E_1/V and E_2/V in the bow in front of "**Convert E/V"** of Analog In1 & Analog In2 sections respectively and set the voltage values as shown in Fig. 7.

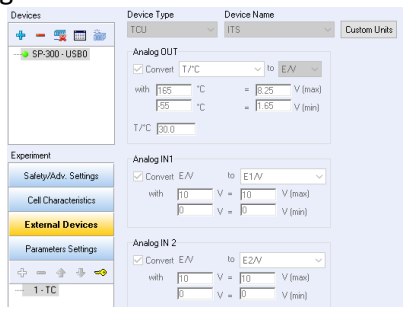

Figure 7: Analog In1 and Analog In2 settings for calibration.

- In Safety/Advanced Setting tab, check "smooth on" box with 10 points.
- In the Parameter settings tab, set the setpoint temperatures in the TC protocol (Fig. 8).

At least two temperature setpoints are needed to plot the linear Temp *vs.* Voltage graph for each Analog input. For a better accuracy three temperature setpoints were used here. Figure 8 shows the Temperature Control "TC" technique with three sequences corresponding to 30 °C, 60 °C and 90 °C setpoint temperatures used for the calibration of the two Analog inputs.

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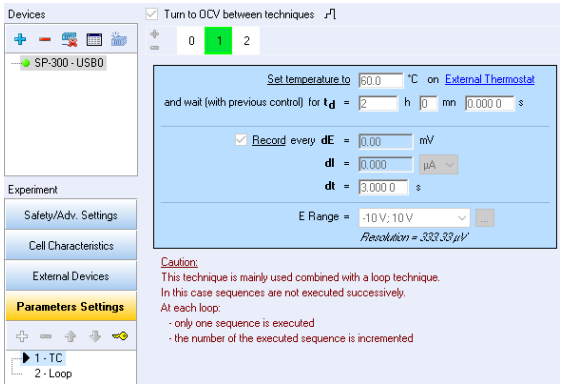


Figure 8: TC protocol with three sequences used for the calibration of Analog In1 and Analog In2.

- Run the "TC" protocol and display the voltage versus time plots.

Once the steady state has been reached, the user has to read and note either the T_{base} and T_{cell} temperatures and their corresponding voltage values measured on EC-Lab®.

Figure 9 shows a picture of the ITS screen obtained with the setpoint temperatures 60.0 °C and 90.0 °C

Figure 9: Picture of the ITS screen showing the steady state values of T_{base} and T_{cell} obtained for the setpoint temperature of 60 °C (left) and 90 °C (right).

The voltage *vs.* time plots obtained with the three setpoint temperatures for analog In1 and Analog In2 are displayed on the Fig. 10.

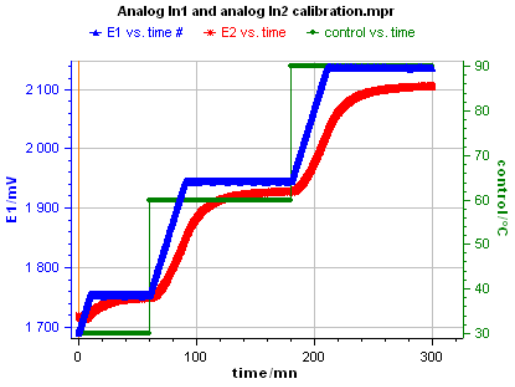


Figure 10: E_1 vs time and E_2 vs. time plots obtained for a simultaneous calibration of Analog In1 & Analog In2.

The measured steady state temperatures (T_{base} and T_{cell}) and voltages (E_1 and E_2) are summarized in the Table II below:

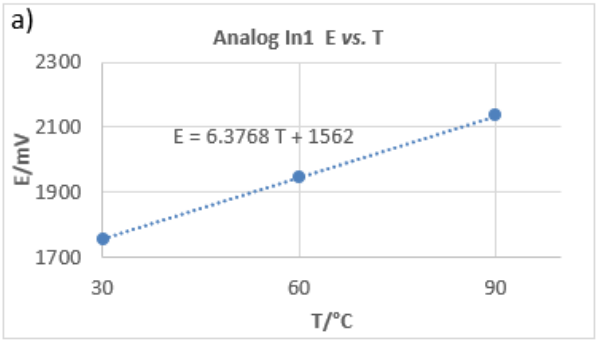
Table II: Summary of the T_{base} & T_{cell} temperatures and their corresponding voltages measured for each setpoint temperature.

ITS setpoint temperature/°C	T_{Base}/°C	T_{cell} /°C	E_1 (Analog IN1) /mV	E_2 (Analog In2) /mV
30.0	31.1	29.4	1754	1751
60.0	60.0	57.8	1945	1931
90.0	90.0	85.0	2136	2106

Note

We strongly recommend you wait until the voltage E_1 (and also E_2) reaches a plateau. This means that T_{base} and T_{cell} have reached the equilibrium temperature. Indeed, T_{base} and T_{cell} displayed on ITS screen correspond to average temperature values whereas the T_{base} and T_{cell} acquired by EC-Lab® are instantaneous temperature values. When the steady state is reached, the instantaneous temperature values acquired by EC-Lab® are constant and are equal to the average temperature displayed on the ITS screen.

The Table II data were used for drawing the Voltage *vs.* Temperature plots of the calibration of Analog In1 and Analog In2. Figure 11 shows the obtained plots.



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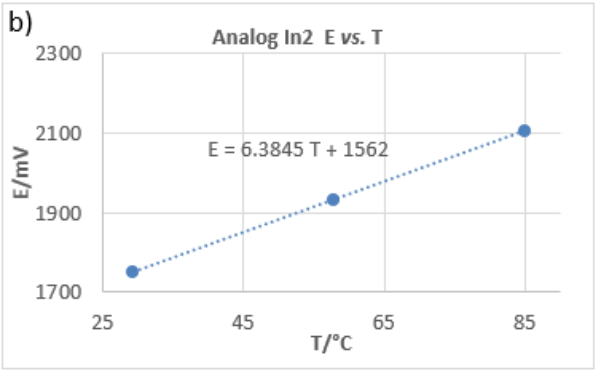

Figure 11: Voltage vs. Temperature plots due to the calibration of the Analog In1 (a) and Analog In2 (b).

The Voltage *vs.* Temperature plots were fitted to straight lines with the equations:
E = 6.3768 T + 1562 for Analog In1
E= 6.3845 T + 1562 for Analog In2

By default, the temperatures - 40 °C, 150 °C and the voltage at both temperatures are used as settings for Analog input in EC-Lab®. They are calculated using the calibration equations above.

Analog In1:

For T_1= - 40 °C; E_1= 1.307 V

For T_2= 150 °C; E_2= 2.519 V

Analog In2:

T_1= - 40 °C; E_1= 1.307 V

T_2= 150 °C; E_2= 2.519 V

These points are set in the Analog In1/In2 sections of the TCU device of EC-Lab®. The setting with these two points has to be checked for at least two temperatures.

V– CONCLUSION

This note showed how a potentiostat user can calibrate analog out, analog In1 and Analog In2 of a potentiostat board used for the control of an ITS. This calibration improves the accuracy of temperature control and the accuracy of T_{base} and T_{cell} acquirements and so leads to reliable temperature measurements.

REFERENCES

1) Operating manual of ITS
2) Technical note #2 CESH Sample holders.

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