

# ON THE ADVANCE OF IMPEDANCE MEASUREMENTS FOR MONITORING MOISTURE IN SANDSTONE

F. Lehmann<sup>1</sup>, M. I. Martínez Garrido<sup>2</sup>, M. Krüger<sup>3</sup>

## ABSTRACT

Electrical impedance measurements may be used to obtain information about the moisture content of porous materials. The impedance of moist sandstone is dependent on its temperature, but also on the pore size distribution and pore volume, as well as other parameters. In this paper we describe the application of an Arrhenius-type temperature compensation approach and the particularities for in-situ compared to laboratory measurements.

## Keywords

Electrical impedance measurements, moisture, salt, cultural heritage preservation, temperature compensation.

## 1. Introduction

Measurements of a sandstone's electrical impedance can be used as an indicator for its moisture content. Although it is currently not possible to deduct absolute moisture values without extensive prior calibration, continuous monitoring can help to trace moisture migration. The two major influences besides the moisture that can change in the sandstone over short periods of time are the salt content and the temperature. While the former is generally an actor that is of strong interest to the surveillance operator, the latter can be regarded as an interfering factor. It is therefore desirable to eliminate the temperature from the impedance readings.

Instrumented impedance and temperature measurements were carried out in the laboratory and at an outdoor facility to evaluate the possibilities of temperature compensation for the used monitoring system.

## 2. Temperature compensation

The temperature can be easily monitored with a wide variety of methods, e.g. using a material's characteristic temperature-dependent resistance (thermistors), the differential voltage between two unequal conductors (thermocouples), or the forward voltage of semiconductor diodes. Within the natural ambient temperature range their readings are very accurate and reproducible.

Electrolytic conductors, as is the case for moist sandstone, can be regarded as NTC-thermistors (negative temperature coefficient). That is, the electrical impedance decreases with

increasing temperature. To compensate the temperature influence in such a measurement, different approaches can be found in literature. They are based on equating the impedance ( $Z$ ) as a function of temperature and some parameters ( $A$ ,  $B$ ) for a reference ( $T_0$ ) and the measurement temperature ( $T_1$ ) via one of the parameters.

$$Z = f(T, A, B) \quad (1)$$

$$\Rightarrow Z_1(T_1) = f(Z_0, T_0, T_1, B) \quad (2)$$

Small temperature offsets may be compensated using a linear or polynomial function (e.g. [1], [2]) as given in equation (3).

$$Z_1 = Z_0 \cdot [1 + \sum_{i=1}^n B_i \cdot (T_0 - T_1)^i] \quad (3)$$

As an often used alternative, Steinhart and Hart [4] propose a partial sum with logarithmic members, which also provides a good fit for thermistors. The extended Steinhart-Hart equation (4) is given below as temperature in dependence on the impedance due to its intricate inverse representation.

$$T^{-1} = \sum_{i=0}^n B_i \log^i Z \quad (4)$$

Yet, most commonly, an Arrhenius equation is employed to relate temperature and impedance (e.g. [3], [6]). Arrhenius [5] examined the connection between the chemical reaction rate ( $k$ ) and the temperature, which led to equation (5). The decoupled equation is given thereafter.

$$k_1 = k_0 \cdot e^{B \cdot (T_1^{-1} - T_0^{-1})} \quad (5)$$

$$k = A \cdot e^{\frac{B}{T}} \quad (6)$$

It is, however, not obvious by itself - except for the obviously similar temperature compensation curves - why the electrical impedance should comply with the same principles as the reaction rate! To explain this we have to regard the definition of the latter, which is the proportion of change in the chemical concentration ( $\Delta c$ ), i.e. the amount of substance ( $n$ ) in a certain volume ( $V$ ), over time ( $t$ ). In analogy, we can define a new variable  $\kappa$  as the ratio of change in the volume charge density ( $\Delta \rho$ ) and time ( $t$ ).

$$k(t) = \frac{\Delta c}{\Delta t} = \frac{\Delta n}{V \cdot \Delta t} \quad (7)$$

$$\kappa(t) \stackrel{\text{def}}{=} \frac{\Delta \rho}{\Delta t} = \frac{\Delta Q}{V \cdot \Delta t} \quad (8)$$

Now, considering that the electric charge ( $Q$ ) is the product of the unit charge ( $e$ ) and the amount of charges, and substituting the according section with equation (7) gives

$$\kappa(t) = \frac{\Delta Q}{V \cdot \Delta t} = \frac{e \cdot \Delta n}{V \cdot \Delta t} = e \cdot k(t) \quad (9)$$

1 Materials Testing Institute University of Stuttgart (MPA Stuttgart), Germany, frank.lehmann@mpa.uni-stuttgart.de

2 Instituto de Geociencias, IGEO (CSIC-UCM) and CEI Campus Moncloa UCM-UPM and CSIC, Madrid, Spain, mi.martinez.garrido@csic.es

3 Materials Testing Institute University of Stuttgart (MPA Stuttgart), Germany, markus.krueger@mpa.uni-stuttgart.de



Combining this equation with the definition for the electric current ( $I$ ) as the quotient of charge and time, and furthermore inserting the electrical impedance yields

$$\frac{\Delta Q}{\Delta t} = I(t) = e \cdot k(t) \cdot V \quad (10)$$

and finally

$$Z(t, t') = \frac{u(t')}{I(t, t')} = \frac{u(t')}{e \cdot k(t, t') \cdot V}, \quad (11)$$

where  $t'$  relates to the periodic alternating current and  $t$  to the time dependence with regard to a change of state. The former may therefore be disregarded for our purposes.

The inverse of the impedance is the conductance ( $\sigma$ ), which replaces the impedance in equation (11). The time independent variables  $U$ ,  $e$  and  $V$  can then be embraced in a constant  $C$ . Merging with equation (6) provides the relation for the temperature dependence of the impedance.

$$\sigma = Z^{-1} = C \cdot k = C \cdot A \cdot e^{\frac{B}{T}} = A' \cdot e^{\frac{B}{T}} \quad (12)$$

$$Z = C^{-1} \cdot k^{-1} = A'' \cdot e^{-\frac{B}{T}} \quad (13)$$

The initially proposed analogy between the chemical concentration and the charge density can also be found in a comparison of the initial energies for constant pressure and temperature. While the chemical energy is defined as

$$G_{chem} = \mu \cdot V \cdot c, \quad (14)$$

the electrical energy is given by

$$E_{elec} = U \cdot V \cdot \rho. \quad (15)$$

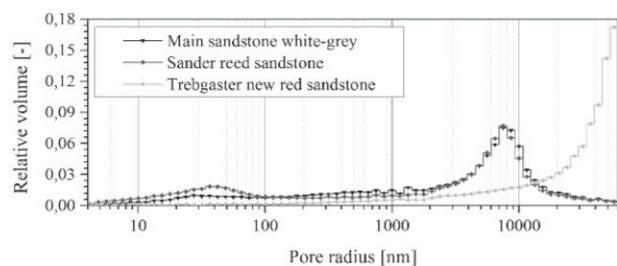
In the above equations (14) and (15), the first term corresponds to the potential, the second to the volume, and the last term to the respective chemical and charged particle concentrations as used in the derivation above.

### 3. Experimental work

To determine the temperature influence on the impedance measurements on different sandstones and for different moisture levels, a test series was performed at the MPA laboratories.

#### 3.1 Methodology

Monitoring and data gathering were performed with a wireless impedance measurement system developed by the MPA University of Stuttgart and TTI GmbH – TGU Smartnote [7] (Figure 2). Three sandstone types with different characteristics were used in the tests: Main sandstone white-grey (M), Sander reed sandstone (S) and Trebgaster new red sandstone (T). Their pore size distributions are given in the figure below.



**Figure 1 Pore size distributions of the used sandstones types. Pore volume: M 18.9 %, S 19.9 %, T 15.6 %; bulk density: M 2.17 g/cm<sup>3</sup>, S 2.10 g/cm<sup>3</sup>, T 2.23 g/cm<sup>3</sup>; free water absorption: M 6.49 m-%, S 7.45 m-%, T 8.76 m-%.**

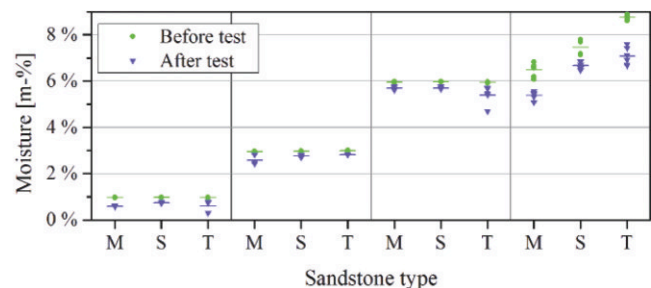
The specimens were cut into cubes with 40 mm edge length. To place the sensors, three holes with 6 mm diameter and 20 mm depth were drilled in the vertices of an isosceles triangle in one side of each specimen. The two holes at the triangle base with a center-to-center spacing of 16 mm (10 mm free spacing) were used for the impedance sensors. One out of three specimens of each sandstone type was equipped with a temperature and humidity sensor in the third hole at a center-to-center leg spacing of 12 mm.



**Figure 2 Wireless sensor node used for the impedance and temperature/humidity measurements.**

A two-point measurement setup with flexible nickel-graphite-silicon sensors as described in [7] was used for the impedance measurements. A frequency range of 10 Hz to 100 kHz was covered with 41 discrete measurements at a logarithmic spacing. The temperature and humidity in the boreholes were determined using capacitive Sensirion SHT25 sensors, which is a version with enhanced precision.

For the initial specimens characterization their weights including and excluding the sensors were taken after drying to constant weight at 60°C. The specimens were then successively loaded with a certain amount of de-ionized water to adjust then to moisture levels of 1, 3, 6 and 9 m-%. The last moisture level was not reached for any of the sandstone types. Instead, the test was carried out with a mean free water uptake of 6.49 m-% for M, 7.45 m-% for S, and 8.76 m-% for T specimens. For the tests with 1 and 3 m-% moisture content 3 specimens of each sandstone type were used, and 6 specimens each for the experiments with 6 and 9 m-% moisture.



**Figure 3 Moisture levels before and after each test.**

After adjusting each moisture level, the specimens were wrapped in foil and stored for at least 24 hours. The water content was then gravimetrically rechecked and the



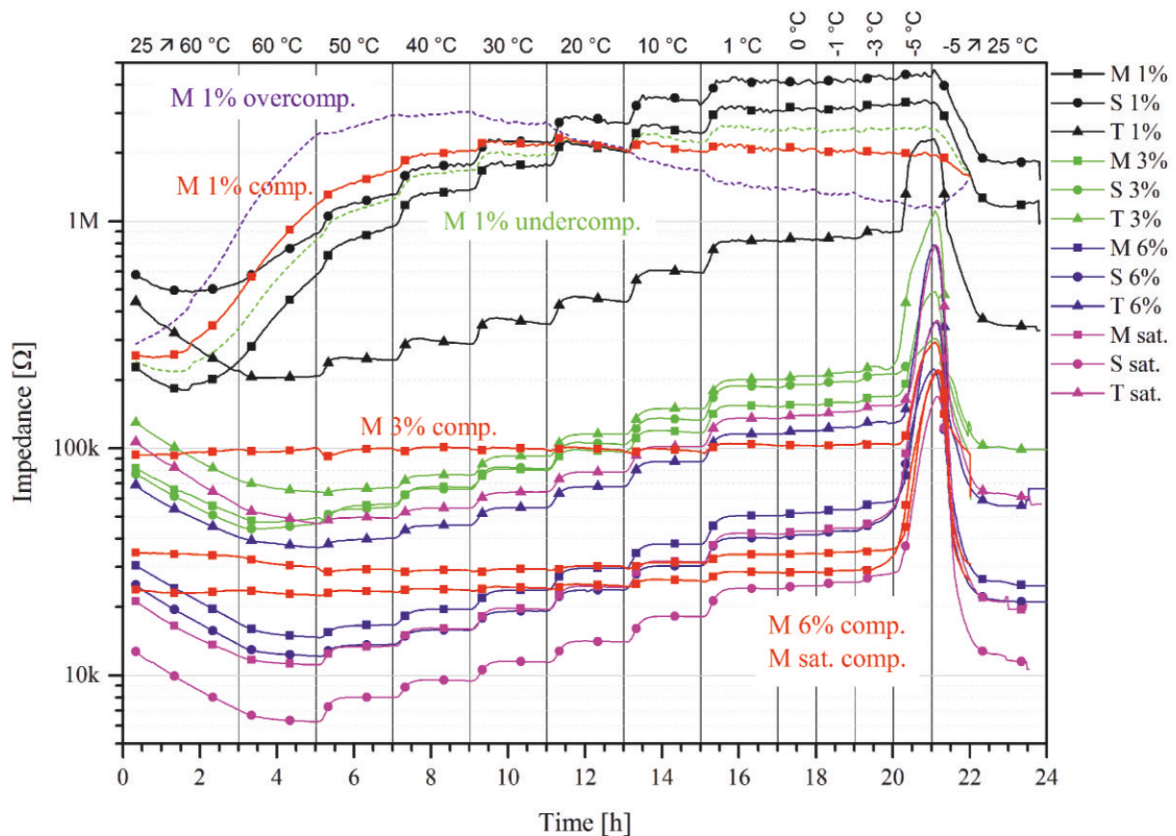
wrapped specimens were placed in a temperature chamber (uncontrolled humidity) and the sensors connected to the measurement system. The impedance was measured at intervals of 5 min, the temperature and humidity inside the specimens every 10 min.

A 24-hour stepwise temperature curve was then automatically operated. During the first three hours, the temperature was raised from 25 to 60°C, followed by a plateau at that level for 1:50 hour. The temperature was then decreased to 50°C within 10 min, which was again held for 1:50 hour, and so on until a temperature of 10°C and then 1°C was reached. The following plateaus at 0, -1, -3 and -5°C were each held for only 50 min due to the smaller temperature decrement between the steps. Finally, the temperature was again linearly raised to 25°C. Following the cycle, the specimens were weighted including sensors and foil in order to determine the water loss during the test. Figure 3 shows the actual moisture content for the M, S and T specimens and their mean values within one sandstone type before and after the test.

### 3.2 Results

In accordance with section 2, the lowest impedance values were obtained for the highest temperatures, following a stepped distribution according to the changes of the temperature cycles (Figure 4). Although the temperature steps were evenly spaced, note that this corresponds to a logarithmic change in the impedance. That is, the temperature influence is considerably higher for lower temperatures, with “lower” being in the range of the natural ambient conditions.

Below -3 to -5 °C, the water freezes, leading to a positive jump in the impedance due to the lack of conductive electrolyte. The freezing point depression may be accounted for by the small capillary radii of the sandstones. In [8], Meller reports depressions similar in value for Indiana and Berea sandstone. He furthermore examines the influence of different moisture contents on the freezing point and describes a decrease of the depression, i.e. a freezing point closer to 0 °C, with increasing moisture. The same effect can be found in our measurements, where the water in the sandstone types with smaller pore radii did not freeze at all in the case of 1 m-% moisture.



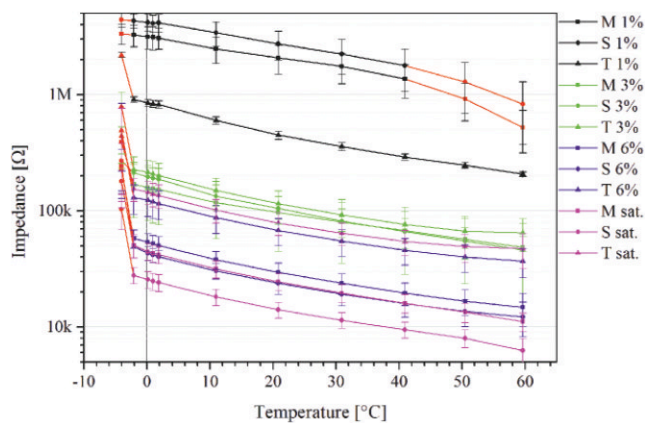
**Figure 4** Development of the impedance at a frequency of 1 kHz over time for 1, 3, 6 m-% and saturated moisture content. The exemplary temperature compensated curves are labeled and marked red.

During the first 5 hours of the test, no constant impedance settled for the M and S specimens at 1 m-% moisture, which is most probably a result of water loss. At generally low water contents, the evaporation of only a small amount of water already changes the percental moisture content significantly. Also, the impedance for the T specimens

measured at saturation seems to be too high during the entire measurement (similar for all 6 specimens), although none of the controlled parameters were out of range. A reason for this could be the different pore space and pore size distribution of the materials.



Compared to the other two materials (M and S), sandstone T inhabits a lower total pore volume, but also a larger average pore size diameter. Consequently, the internal pore surface is less and thus the adsorption behavior and water distribution inside the pores is different. It is known that the conductivity of water in porous media is only given for water that forms multilayers, so it seems that the amount of capillary and adsorbed water inside the pores is to be considered in the analysis. However, this aspect is part of further studies.



**Figure 5 Impedances for each sandstone type and moisture content in relation to the according temperatures.**

The simultaneous measurement of impedance and temperature allows the deduction of a relationship between both parameters. For this purpose, a mean value for the impedance during the final 30 minutes of each temperature plateau was calculated. Figure 5 shows the impedance for each of the sandstone types and moisture levels plotted against the according temperatures. As expected from theory, the relationship follows an exponential decrease function.

**Table 1 Parameter  $B$  in (5) for  $A = 100 = \text{const.}$**

	M	S	T
1 m-%	2851	2919	2491
3 m-%	2023	2057	2091
6 m-%	1692	1635	1934
saturated	1626	1468	(1981)

Keeping  $A$  in (6) constant, the parameter  $B$  for use in (5) can be calculated as given in Table 1. Four exemplary compensated curves are shown as red lines in Figure 4 using the determined values for  $B$  given in the table. The impedance becomes a horizontal linear line as desired, except for the 1 m-%, for the reasons given above.

### 3.3 Application to in-situ measurements

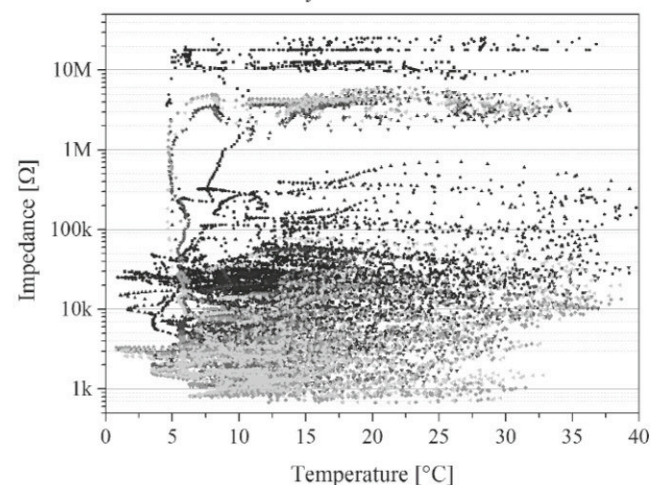
In laboratory conditions with only one varying parameter, e.g. temperature or moisture content, it is, as shown, possible to compensate for the temperature influence. However, under in-situ conditions, generally both parameters are varied together (it gets cooler when it starts raining, etc.). Noting the strong variation of  $B$  for different moisture contents in the table above illustrates the emerging difficulty.

Although  $B$  can be calculated from laboratory tests for different moisture levels, the application of (5) using a “correct” parameter  $B$  to data where both influences are varied is not possible. Choosing some arbitrary value for  $B$  will therefore inaccurately compensate for the temperature. In Figure 4, the impedance curves for 1 m-% moisture are exemplary compensated using a value for  $B$  that is too high (purple dashed line) and too small (green dashed line). For the case of  $B$  being too high, the impedance is overcompensated for temperatures higher than the reference, and undercompensated for lower ones.

Although this does not necessarily rule out temperature compensation in general, it is a very important finding for on-site impedance measurements and has two implications. On the bad side, if the temperature cannot easily be compensated, much care has to be taken when analyzing the data regarding moisture. On the other hand, if the relationship between impedance and temperature is unclear for in-situ measurements in such a way, some other parameter - the moisture - plays a role just as important. In fact, the temperature variation within one moisture regime is even quite small compared to the moisture influence.

To test the temperature compensation, two sandstone specimens were placed at a natural, freely weathered environment. Specimen 1 was a cornice piece of a church roof (medieval Heimbacher sandstone from the Minster Freiburg), well away from all artificial salt sources. Specimen 2 was from the base of a historic door (Trichtinger sandstone, Rottenburg).

Figure 6 shows the impedance versus the temperature data recorded over a time period of two months. It quickly becomes clear that it is not meaningful to do a curve-fitting on the data as described before. Hence, a value for  $B$  of 2000 was freely chosen and temperature compensation according to (5) was carried out. The original and compensated data is shown in Figure 7. Additionally, the precipitation height (blue crosses) and sunshine duration (yellow circles) are indicated as relative values. It is striking that, although the temperature is compensated, the impedance variation is still in the order of magnitudes, which is in agreement with the statements before. Also daily fluctuation could be observed.



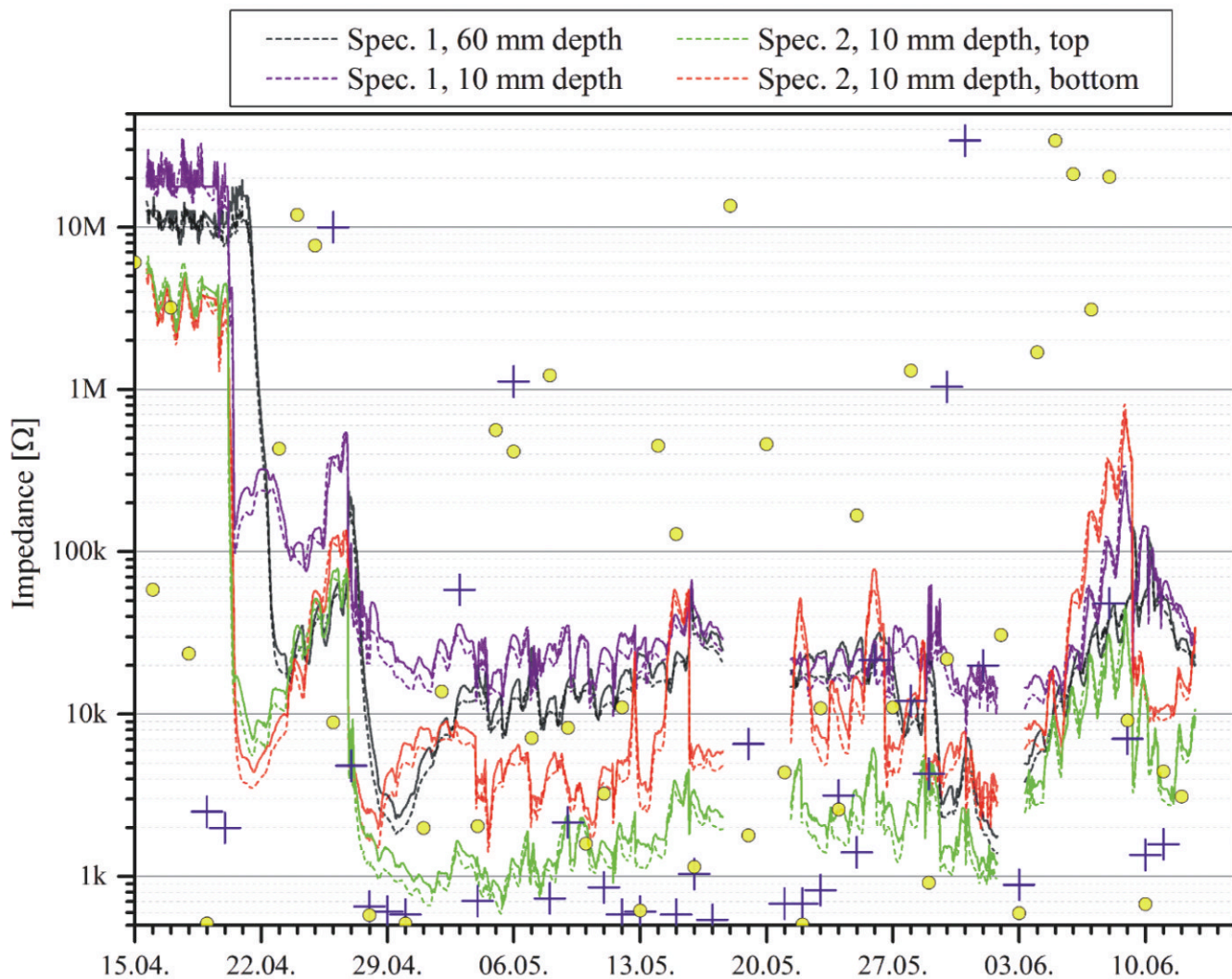
**Figure 6 In-situ measurements of impedance versus the according temperature. Each shade of grey corresponds to a sensor.**



The analysis is quite straight forward. On 16<sup>th</sup> April, the specimens were placed outside after being stored in a dry place. Accordingly, the impedance at this point was very high. The strong drop in the data is due to the first rainfall on 19<sup>th</sup> April, providing an electrolyte and hence significantly changing the specimen's conductivities. The time offset between the rainfall and the response of the impedance is due to the time required for the water to reach the sensors. This is especially clear for the deeply placed sensor in specimen 1, which only responds with an additional delay of about 2 days. During the following rainy period, the impedances for all sensors show only little fluctuations. It can be concluded

that the sandstones are saturated and have reached their final, i.e. lowest possible impedances. The still comparably high absolute value of 10 k $\Omega$  in specimen 1 indicates a low salt concentration and no salt migration within the stone. Specimen 2 probably has a higher salt concentration, which is visible in the low impedance of 1 to 5 k $\Omega$ . Being a sandstone from a doorway, this is quite possible.

On the 3<sup>rd</sup> of June, a short sunny period started with long sunshine duration and no rain. The sandstones started drying, which consequently was accompanied by a rise in the impedance.

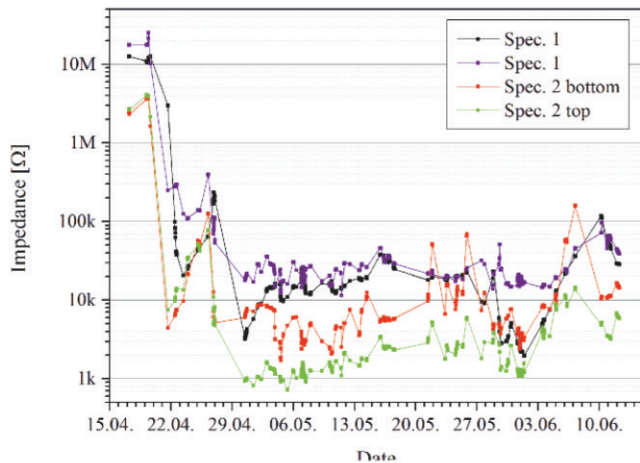


**Figure 7** Temperature compensated development of the impedance for a reference temperature of 20 °C and a parameter  $B$  of 2000. The dashed lines are the original data, the solid lines the compensated curves. The blue crosses indicate the precipitation height, the yellow circles the sunshine duration (data by courtesy of the German meteorological service DWD). The weather data is scaled to fit the graph and indicates only the relative changes.

The temperature compensation using arbitrary values for  $B$  may be unsatisfactory due to the somewhat erroneous results. One great advantage of instrumented monitoring can be the large amount of data that is gathered. In the case in hand, to be able to deduct conclusions about the moisture development in the specimens, we can reduce the data to areas within a certain temperature range. A reasonable

choice would be some temperature that is often crossed in the considered time period, i.e. the median temperature. For the present data this would be 12.4 °C. To enlarge the dataset, a temperature range between 11.4 and 13.4 °C was chosen. Figure 8 shows the selected impedance set for six individual sensors. The dots mark the single measurements.





**Figure 8 Development of the impedance at the outdoor facility for the 11.4 to 13.4 °C temperature range.**

Note that this method is only helpful if long-term data analysis is to be done. By excluding measurement points the short-term data quality necessarily suffers.

However, by elimination of the temperature influence through removal of all points outside a fixed range, the remaining fluctuations are due to changes in the moisture only. The curve progression confirms the compensation of the data in Figure 7.

#### 4. Conclusions

The temperature influence on electrical impedance measurements on sandstone follows an exponential decrease function in the form of an Arrhenius equation. It can therefore be compensated accordingly.

However, it must be qualified that the temperature compensation function is also dependent on the moisture level of the regarded material and the water distribution inside the pores, i.e. the amount of adsorbed and capillary water. That is, for a temperature compensation the material moisture has to be known, which is generally not the case. And furthermore, if both temperature and moisture are variable over time, the compensation function for the temperature varies with the development of the moisture itself. This back-coupling of the temperature compensation function with the material moisture hence eliminates the possibility of mathematically separating both parts.

Certainly the parameter for the moisture in the temperature compensation function may be chosen freely. It has then to be taken into consideration that the compensation is most likely done with a wrong assumption for the moisture content. Yet, when long-term impedance data is to be evaluated, the fluctuations due to the moisture changes are much larger than from the temperature and may therefore not play a major role.

#### 5. References

- [1] Standard ISO 7888:1985. Water quality. Determination of electrical conductivity. International Organization of Standardization (ISO). Adopted as European standard EN 27888:1993.
- [2] Standard IEC 60751:2008. Industrial platinum resistance thermometers and platinum temperature sensors. International Electrotechnical Commission (IEC). Adopted as European standard EN 60751:2008.
- [3] W.J. McCarter. 1995. Effects of temperature on conduction and polarization in Portland cement mortar. *Journal of the American Ceramic Society*, 78(2):411-415.
- [4] J.S. Steinhart and S.R. Hart. 1968. Calibration curves for thermistors. *Deep-Sea Research*, 15(4):497-503.
- [5] S. Arrhenius. 1889. Über die Reaktionsgeschwindigkeit bei der Inversion von Rohrzucker durch Säuren. *Zeitschrift für physikalische Chemie*, 4, 226-248.
- [6] Standard IEC 60539-1:2002. Directly heated negative temperature coefficient thermistors. Part 1: Generic specification. International Electrotechnical Commission (IEC), 2002. Adopted as European standard EN 60539-1:2008-11.
- [7] F. Lehmann, M. Krüger. 2011. Wireless impedance measurements to monitor moisture and salt migration in natural stone. In: Markus Krüger (ed.), *Cultural Heritage Preservation, European Workshop on Cultural Heritage Preservation*, Berlin, Germany, Fraunhofer IRB Verlag, 224-231.
- [8] M. Meller. 1973. Mechanical properties of rocks at low temperatures. In: *Permafrost. North American contribution to the second international conference*. Yakutsk, U.S.S.R. ISBN 0-309-02115-4.