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Time to failure of electric cables in seawater

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Time to failure of electric cables in seawater

Task:

Time to failure of offshore communication cables is an important parameter for ~~economic~~ development of offshore wind farms and for electrification of offshore oil and gas units. Furthermore, the subject also influences all subsea developments in the O&G industry.

The time to failure depends on the sheath surrounding the cable core and the temperature of the water as well as voltage and the external water pressure.

In this thesis, the student will investigate state of art design of communication cables and carry out experiments for selected parameters to determine the adaption of the laboratory results to the lifetime of cables in the field.

These parameters to be investigated are sheet material, salt content and seawater temperature.

A limited study of the influence of the pressure gradient would also be an interesting extension of the scope. It will be investigated if such tests could be done at the University of Rostock.

Abstract:

In this paper, the standardized insulation resistance test in water for predicting the service life of low-voltage cables is investigated. The basic explanations for determining thermal ageing and estimating the service life according to DIN EN IEC 60216 are based on the assumption of a first-order kinetics of the thermal decomposition reaction of the insulation material. Thermal oxidation, pyrolysis and hydrolysis of the base polymer are among the most important degradation reactions of an insulating material. Hydrolysis reactions are higher order reactions, since the reaction rate is determined by the concentration of oxygen or water.

A methodical approach is presented using the example of an iteration loop to optimize the prediction of the time to failure. The non-destructive electrical method, the insulation resistance test is used as the tool for determining a reference value. Therefore, uncertainty in the measuring method for insulation resistance measurement is evaluated. The precise determination of an insulation resistance as a limit value is described and subsequently the change in the insulation resistance of thermally aged and actually aging cables is approximated to a material constant and a temperature function with the aid of an adjustment function. On the basis of this, the material could be classified and evaluated, since the indication of the numerical value of the insulation resistance is only of limited value, since it is very strongly dependent on the measuring conditions.

Inhalt

1 Introduction

"The dimension of an insulated wire or cable are functionally important because the safe operation of the circuit depends on an adequate amount of insulation. Design engineers make use of the dimensions to ensure that adequate space is allowed for the wires and cables in the packageing of the circuit." (ASTM D 3032, p. 13)

This expression contains an oxymoron. The type of insulation and the space, both cost money. The constructive solution is driven by economic factors. And in the field of offshore constructions space is associated with high cost. "The offshore industry places extremely high demands on offshore cables. Offshore cables need to be of very high performance while simultaneously facing harsh environments and aggressive chemicals. There are also high safety demands being placed on offshore cables." (Vries 2014, p. 4) For example subsea production systems can be used to develop reservoirs, or parts of reservoirs, which require drilling of the wells from more than one location. Deep water conditions, or even ultradeep water conditions, can also inherently dictate development of a field by means of a subsea production system, since traditional surface facilities such as on a steel-piled jacket, might be either technically unfeasible or uneconomical due to the water depth.

„For the purpose of describing the various flowline and umbilical components, it is convenient to divide them into lines that convey fluids, i.e. pressure containing lines, and lines that do not convey fluid, i.e. electrical and fibre optic cables. […] Separate electrical cables may also be required for transmission of control signals/data in an electrohydraulic PCS. Alternatively, the control signals/data may be superimposed on the power output, commonly referred to as -signal on power-". (DIN EN ISO 13628-1, pp. 140–141)

This means that operation must be guaranteed at all time. The service life[1] of a cable depends on the quality of the insulating material. "Over the years, however, the quality of these insulating materials decreases due to stress and environmental influences." (Chauvin Arnoux, p. 2) According to Koldrack (Koldrack 2009, p. 13) the environmental influences can be subdivided:

- chemical: small size of the water molecules facilitate penetration --> hydrolytic degradation (penetrating water leads to splitting: polymers -->oligomers -->monomers) lower molecular weight
  redox active copper ions (Cu^+ and Cu^{2+} act as catalyst in the degradation of polymers
- physically: Expansion due to uneven heating; fixed and flexible cables have a frictional load; radiation loads
- biological: Microorganisms use polymer materials as food

Load types can act individually and/or in combination on underwater cables and superimpose each other in their effect. For practical use, individual types of load should be known. In order to assess the service life of a cable, the insulation properties and application limits must be determined by a valid measuring method, i.e. non-destructive measurement of insulation resistance.

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[1] Definition according to Rudorf "Service life - Time required to reduce a pre-defined property of a material by a defined percentage or to a defined final value under the influence of one (or more) selected load(s)" Rudorf, p. 131.

1.1.Initial situation

Type approvals by classification societies for the offshore cables industry (Lloyd's Register, Bureau Veritas and DNV GL) guarantee that quality is being maintained throughout the whole production process. Cables are tested according to the existing industry standards, and are certified when it is established that these standards are met. (Vries 2014, p. 8) According to Benedict van Oosterbosch, Incore Cables, the offshore cable industry uses four types of standards worldwide. These standards are referred to are called: ASTM D3032, VG 95218, the NEK 606 and IEEE 1580. These standards are relevant for cables on the topsides and production platforms and FPSO's. They mainly relate to the conductor sizing, screening, insulation and sheathing compounds. These standards are limited for subsea applications and will be investigated in chapter *2.3 **Fehler! Verweisquelle konnte nicht gefunden werden.***.

Recommended practice for insulation properties is testing insulation resistance. In the test procedure a high DC voltage will cause a low current (in the order of 10^{-3} A) to flow through the conductor and insulation. The amount of current depends on the amount of voltage supplied, the capacity of the system, the total resistance and the temperature of the material. The total resistance is the sum of the internal resistance of the conductor and the insulation resistance in Ω. There are various methods for testing non-destructive measurement of insulation resistance:

Point measurement testing, also short time measurement is the most frequently used method. The test voltage is applied for a defined period of 60-120 seconds. This method returns a time/resistance curve that cannot be considered an absolute value for the insulation resistance. The value depends on the time at which the measuring point was measured, and it varies with the time span. In addition, factors such as temperature and water composition influence the test results.

Tendency from point measurement, by comparing periodically determined point measurement values it is possible to recognize trends. In addition to statements regarding defined limit values that are considered "safe", this allows observations on changes in the condition of the insulation material over time.

Dielectric absorption behaviour (DAR), after applying the test voltage the capacitive charging current moves relatively quickly to zero. However, it takes some time (possibly hours) until the molecules of the dielectric are polarized and the absorption current drops. In addition, a leakage current occurs which superimposes the change in the charging currents.

Over time, the curve of insulation resistance increases and flattens out. The dielectric absorption ratio can be represented by point measurements at fixed times and the derived ratio. The comparison times are usually 30 and 60 seconds. As a result, the measured values are significantly less affected by external influences such as temperature changes than point measurements at intervals of several hours.

Polarization index (PI) to describe the state of the insulation material, the "mobility of the molecules" must be represented more precisely. To do this, the time interval between the comparison points must be increased. For the polarization index, the measured value is set in proportion to the value of one minute after ten minutes. By applying a direct current for a longer period of time, the molecules are collectively aligned and the polarization index, as an extended method of DAR testing, represents the polarization with a ratio value.

A test sequence of different test methods provides different information about the condition of the insulating material. This primarily requires knowledge of the components of the test current. A detailed discussion of the components takes place in *2.2 Insulation testing.*

„The purpose of the test is to verify the integrity of the electrical distribution system. The test sequences should follow a strategy of verifying any subsystem prior to a subsea connection operation. The final test then verifies full integrity, from platform to control module." (DIN EN ISO 13628-1, p. 65) This standard expresses the dilemma. The cable is examined very carefully prior operating. Because of the limited access possibilities monitoring is spartan. For this reason and because the failure is very expensive, the dimensions of the cable sheath are given a large safety margin. In addition, cables have an expected service life, according to Arrhenius behaviour, which in this context is a ballpark figure.

The basic principles of accelerated ageing, based on the Arrhenius behaviour, for determining the time to failure are based on the implicit assumption of a first-order kinetics of the thermal decomposition reaction of the insulation material. Only under these conditions is the long-term thermal diagram linear and a forecast feasible. Rudorf (Rudorf 2016b, p. 48) defines the following steps for the transferability of short-term behaviour at elevated temperatures to long-term behaviour:

- determination of the medium (in which the long-term behaviour is to be determined)
- determination of the parameters on which the long-term behaviour is to be defined
- determination of a basic characteristic curve for the long-term behaviour at the operating temperature, or maximum permissible operating temperature
- determination of the temperature range in which short-term ageing is to be carried out
- execution of short-term storage and testing of the possibility of determining a mathematically representable dependency (Arrhenius behaviour)
- comparison of the measured values determined in the short-term storage and the dependency that can be displayed with the corresponding measured values from the basic characteristic curve

1.2. Formulate the goal and the method

Most standards assume that accelerated ageing processes could be transferred almost universally by means of Arrhenius processes. However, scientific studies refute this claim for chemical processes of a higher order than one. Therefore, the quality of the time to failure forecast must be adjusted, which is directly related to the method carried out according to (DIN EN 60216-6) for polyurethane. This part can only be investigated theoretically and confirmed by secondary analyses. A goal will be:

- To establish a mathematically representable dependency of short time for elevated long-term behaviour

In this context improving testing insulation resistance is of central significance. Measurements in water are predominantly of a unique nature and have not yet undergone sufficient assessment. The aim of the work is to make the measurement results transferable. In previous tests, the electrical insulation resistance was determined according to the known method (DIN EN 50289-1-4). Great uncertainties have arisen in the process. It will be investigated to what extent the following parameters influence the measurements:

- Water composition (change over time)
- Temperature-coefficient α

- Degree of influence of surface leakage current
- Point in time for a base value
- Mutual influence of consecutive measurements due to insufficiently relaxation time

The measure instrument, the megaohmmeter "Megger" has an error of 5 % to 100G Ohm and 20 % above. Testing insulation resistance should be executed in such a way that the value 100G Ohm is not exceeded during measurements, if possible.

- Determining factors for a test set-up

In addition, the water changes. This means that the polarisation index cannot be used because the water molecules are permanently reorienting themselves. It would also be an interesting extension of the scope to elaborate the influence.

- Extent of polarisation

Further adaptation of the laboratory results to the service life of the cables in the field. Therefore, the following parameters and their effect on the insulation resistance are of interest.

- pressure
- sheathing material
- seawater temperature
- salt content

1.3. Structure of the work

One starts at the bottom and defines the aging process. Concentrating on the chemical and physical aspects and leaving biodegradation aside, because of its complexity. From there, the reason for the parameter of choice, the insulation resistance, becomes clear. Then the non-destructive method for measuring the insulation resistance will be examined. This knowledge is the basis for evaluating the method, to predict the time to cable failure. In the standards the limiting values and requirements are defined. However, with one massive restriction. The artificial aging was carried out in the past in air. To establish a standardisable method the standards will be assessed in detail. For the transferability of artificial ageing into the medium water, the relevant terms are defined and explained according to the current state of science. Parameters that are not clearly defined in the standards are examined individually and, if necessary, confirmed by experiments. The adaptation function developed by Rudorf is evaluated and updated. And finally, the method of measuring the insulation resistance can be evaluated and the quality of a forecast can be assessed.

2 Basics and state of the art

A regular survey carried out by Cigré (Technical Brochure 379) revealed an overall failure rate of 0.12 % for 7100 km quantity of cable, after analysing data relating to the installed quantities of AC and DC underground and submarine cable systems. Out of the total of 852 failures, only 49 are not connected to accessories, and among these 49 ,more than 30 are associated with external damage. As Jamie Neilson from Baltic diver says: "less than 1% of the failures are caused by the cable itself". In the study of Cigré all of the internal faults were reported, having no clear correlation with age.

Evaluation of trend of age regarding failures								
Type of Cable	Age at time of failure (Years)							Total
	0 – 5	6 – 10.	11 – 15.	16 – 20.	21 – 25.	26 - 30.	≥ 30	
AC - XLPE	2				2			4
AC - SCOF	4	3		1	5	3	5	21
AC - HPOF					2			2
DC - MI	5	2	2	3	3	2	1	18
DC - SCOF	1	2					1	4
Grand Total	12	7	2	4	12	5	7	49
	24%	14%	4%	8%	24%	10%	14%	100%

Figure 1: Evaluation of trend of age regarding failures concerning the cable itself (Technical Brochure 379, p. 63)

A Reason for this might be the significant changes, which have been made over the past three decades in terms of the introduction of new materials and technologies.

In the field of standardized test procedures, the service life of electrical insulation materials is predicted by determining the temperature index (TI). The focus is on the series of standards DIN EN 60216 and its counterpart ASTM 3032, which describe how thermal ageing is supposed to be carried out to predict the service life. All tests with thermal ageing follow a similar concept. The process simulates ageing in circulating air heating ovens or heating cabinets by storing the specimen at different temperatures above the operating temperature. With the method of non-destructive electronic testing, the changes are determined. Before storage begins at an elevated temperature, the insulation resistance is measured under normal conditions, this value is referred to as the reference. In addition, a fraction of this value is defined to be the limit value, 50 % by default. During the test, the continuous measured values are recorded and compared to the reference. The time at which the limit value is reached will be plotted for different temperatures (see Figure 4). A regression line is generated from at least three such points. The correlation between slope and intercept is based on the Arrhenius approach, according to which there is a linear relationship between the ageing rate and the temperature.
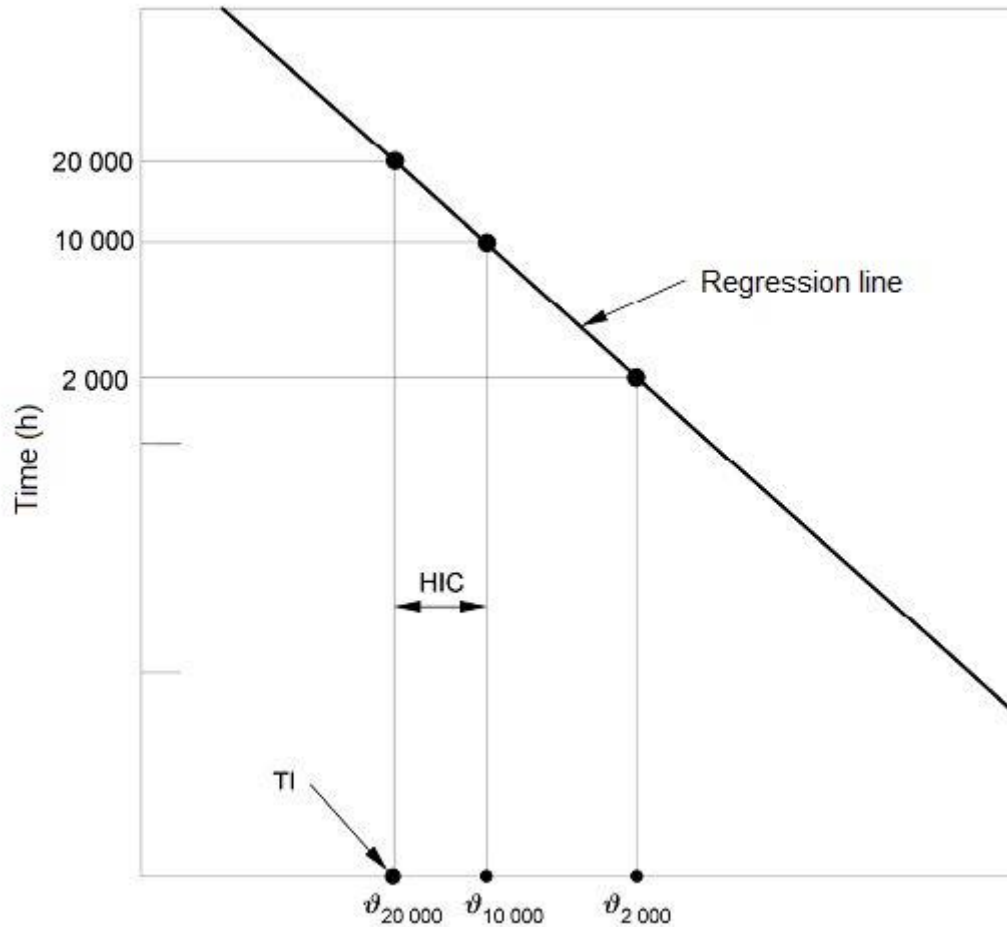
Figure 2 Thermal long-term diagram-with regression line for the determination of the temperature index (20 000 h) (DIN EN 60216-8, p. 14)

The temperature index is determined by extrapolating the regression line to the time of 20.000 h. This point determines the end of service life and the temperature at which the limit value is expected to be exceeded.

There are shortcomings in the application of this test method with the medium of water. Deroine (Deroiné et al. 2014) used this method to investigate the degradation of PLA in seawater. It was found that there was a shift of the degradation mechanism above 40 °C. As for the cause, it was speculated that the main origin of the shift was plasticization. This created additional difficulties in predicting service life. The shifted degradation rate was attributed to a number of possible explanations: osmotic cracking, the formation of diffusion paths induced by cracks, or hydrolysis products such as carboxylic acids with a strong water affinity.

With respect to the main differences in the application of Arrhenius similarity for air and water in the field of accelerated ageing there are different approaches regarding the interpretation of test results. The basis of the degradation mechanisms is formed by physical and chemical processes during ageing. The different order of chemical reaction is identified as the main inadequacy for the transfer of artificial ageing processes. In addition, a relatively high measurement error in the insulation resistance measurement contributes to the relevant inaccuracy of the prediction.

Cigre states that at present a condition assessment only usually results in a "risk of failure" and not a time to failure. To cite chapter and verse:

"The accuracy of the determined risk is related to the following:

1. Reliable knowledge of the history of the given system (operation, failure statistics, changes in the environment, etc).
2. Knowledge of ageing of similar components under similar operation conditions. This knowledge could, for example, be gained from cable system components being investigated in the laboratory after being removed from service.
3. The accuracy of the diagnostic measuring technique.
4. The interpretation of the measured data."

(Technical Brochure 279, p. 62)

Among these factors, the precision of the measuring technique is not the most significant factor. The most relevant factor is most likely to be the interpretation of the measured data for the valid reason, that there is no objective method of verification yet.

The standards associated with insulation resistance and artificial ageing provide in some cases contradictory instructions or recommendations. These are not directly recognizable, due to complex references and multi-level links. As a result, interpretation of standardized test data is not uniform. The current scientific findings are briefly presented in the upcoming chapter. They have the intention to define the frameworking conditions for insulation resistance measurement.

2.1 Process of Ageing

In Domininghaus (Domininghaus et al. 2005), the process of ageing of a polymeric material is regarded as a change in its mechanical, optical and/or electrical properties over time. The ageing of polymers is a complex phenomenon caused by chemical, thermal or mechanical degradation of the material, mostly a combination of these factors takes place. The most important environmental influences that influence ageing and material degradation are:

- Temperature
- Composition of water
- Irradiation by the sun

Temperature mainly influences the kinetics of ageing mechanisms, while other parameters determine the type of mechanism. Moisture, radiation and oxygen encourage hydrolysis, photolysis, photo- and thermo-oxidation reactions, which generally degrade the chemical structure of polymers. Ageing can be divided into two categories: physical and chemical ageing.

Physical ageing

Physical ageing comprises the various processes resulting from the inherent instability of the amorphous phase of polymers. Even in the solid state, the molecules that form this phase are not in thermodynamic equilibrium. The molecules are never held in a certain position at any time, instead they move continuously to reach a more energy-efficient configuration. As time progresses, the relaxation time of amorphous polymers increases. This also contributes to an increase in the distance between the glass transition temperature[2] and the temperature at which the effects of physical ageing are observed. In semi-crystalline polymers, ageing can lead to processes such as secondary crystallization and recrystallization. The effects of physical ageing are often reversible, as it is sufficient to heat the polymer above its glass transition temperature to restore its original properties. (Lechner et al. 2010, p. 58).

Chemical ageing

Processes taking place during chemical ageing lead to a change in the chemical structure of the polymer. This aggregates a large number of reactions of different kinds, whose effects on polymers can even be diametrically opposed. In general, the basic phenomenon underlying almost all chemical degradation processes in polymers is chain scission. The primary chain scission process is the fracture of a bond along the macromolecular chain. In order for this chain scission to occur, the material must be exposed to a certain amount of energy. This can come from different sources: Heat, radiation, mechanical stress, etc. (Kroschwitz 1990).

Sing and Sharma (Singh and Sharma 2008) have categorized the main mechanisms that lead to altered polymer properties as photooxidative, thermooxidative, ozone-induced, mechanochemical,

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2 The glass transition (T_g) of a plastic material is defined as "the reversible transition of the amorphous phase from a rigid, brittle state into a visco-elastic or rubber-elastic state. The glass transition takes place in a more or less wide temperature interval, depending on the hardness and amorphous content of the material respectively. The higher the amorphous fraction, the lower the glass transition temperature and the smaller the temperature interval." (BASF Polyurethane GmbH 2017, p. 10.).

hydrolytic, catalytic and/or biological. Among the various types of primary reactions of chain scission process are homolytic cleavage and heterolytic cleavage, which will be briefly presented.

Homolytic cleavage can be triggered by absorption of radiation (by absorbing the UV spectrum of sunlight) or by thermal systems (in this case binding with lower dissociation energy is most likely). Covalent bonds of two atoms are cleaved by external influences. One binding electron remains with each of the previous partners. Reactions in a non-polar environment produce radicals that are unstable and tend to participate in subsequent degradation reactions (Arriate et al. 2016). In the polar environment, on the other hand, the formation of ions is favoured.

Another primary reaction is heterolytic cleavage. This type of fracture occurs when the material is brought into contact with corrosive liquids or gases. Polymers synthesized by condensation reactions and containing heteroatoms are most strongly affected by ion rupture. Depending on the polarity of the medium, the trigger can generate an ion pair (in a medium with a low dielectric constant) or free ions.

Secondary reactions are generated by the resulting radicals and ions. These include intramolecular reactions (self-reactions) in which macromolecules containing alkyl radicals can combine to form a longer molecule, a branch or a node (Arriate et al. 2016).

During hydrolysis, a hydroxyl group with a proton is split off. The resulting carbon double bond leads to chain scissions and thus to changes in the structure. Due to the higher complexity of the water molecule compared to the oxygen atom, the reaction is of a higher order than for oxidation reactions.

Oxidation reactions rank among the chemical reactions with an order greater than one - or pseudo-first order. They are the predominant degradation mechanism for a significant number of polymers. In general, oxidation is a chemical reaction where electrons are transferred directly between molecules. This transformation causes consecutive reactions. Radicals with carbon atoms as a base, such as polyurethane, react with the oxygen present in the environment to form peroxyl radicals. These in turn attract hydrogen from adjacent molecules to produce a new peroxyl residue and so on. This cycle of radical formation and the removal of hydrogen atoms leads to a self-feeding chain scission. Peroxyl radicals finally stabilize themselves by abort reactions with other radicals to chemical groups such as dialkyl peroxides, carbonyl groups or alcohols (Richaud and Verdu 2002, p. 14).

Chemical cleavage of macromolecules results in changes in the chemical structure, physical properties and appearance of a polymer, independent of the mechanism of chain scission.

For a comprehensive presentation, further degradation processes that can change the long-term behaviour of polymers under certain conditions are added:

- Metal-catalyzed degradation processes: Impurities or traces of additives or pigments of metallic origin (e.g. copper oxide) which are incorporated during polymer processing can accelerate oxidative degradation processes if they are triggered by thermal or chemical agents (Kroschwitz 1990).
- Crosslinking and branching of polymers: Although these reactions are generally harmful because they reduce the elongation and flexibility of polymers, in some cases (as with polyethylene) they are deliberately triggered to increase the processing and softening

temperature. At high temperatures, cross-linking is counteracted by chain scission reactions (Kroschwitz 1990).

- Ozonolysis: Atmospheric ozone mainly attacks polymers containing unsaturated bonds such as rubbers. The harmful effects of ozone exposure on rubber are expressed by the loss of mechanical properties and the formation of surface cracks (Duchateau 1995).

- Degradation by ionizing radiation: Under anaerobic conditions exposure to ionizing radiation (X-rays, –, α, γ, β) leads to different types of reactions depending on the dissociation energy of the C-C bond in the macromolecular skeleton. (Duchateau 1995).

To conclude, all the processes mentioned above are concealed behind the changes of the material property which can be measured:

Mechanical properties:

Chemical degradation processes that lead to a reduction in molecular weight, e.g. chain scission is connected with a loss of mechanical properties. Due to ageing, properties with elastic deformation, such as the E modulus, remain virtually unchanged. In contrast, the influence of ageing has an effect on permanent properties such as breaking elongation. In unoriented linear polymers, macromolecules lose their ability to form nodes when they fall below a critical threshold. This leads to a significant decrease in the elasticity of the polymer. In this case, polymers with originally ductile behaviour undergo a transition to brittle behaviour (Domininghaus et al. 2005).

Optical properties:

Some degradation products may have absorption spectra in the visible range that lead to colour changes (Koldrack 2009, p. 83).

Electrical properties:

The products of chemical degradation reactions, including oxidation reactions, are often polar groups that can change the dielectric behaviour of the material by introducing new dissipation bands. This causes an insulation property that changes over time. Oxidized groups can also cause a decrease in dielectric strength (Arriate et al. 2016, p. 8).

This paper uses the latter electrical properties as an indicator for the degradation process and uses the non-destructive measurement of insulation resistance to assess the ageing of polyurethane cables.

2.2 Insulation testing and influencing factors

„A number of factors affect the value of the insulation resistance and therefore the value of the current flowing when a constant voltage is applied to the circuit being tested. These factors, such as temperature or humidity for example, may significantly affect the measurement result." (Chauvin Arnoux, p. 3). There are various methods of insulation resistance testing, which are briefly presented below.

Short-time or spot reading measurement, is the most frequently used method. The test voltage is applied for a defined period of 60-120 seconds. This method returns a time/resistance curve that cannot be regarded as an absolute value for the insulation resistance. The value depends on the time at which the measuring point was measured and varies with the time span. In addition, factors such as temperature and water composition influence the test results.
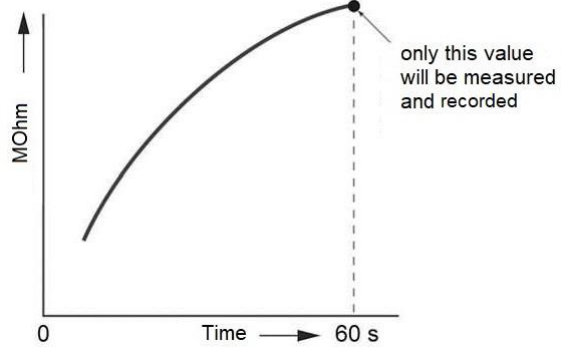
Figure 3 short-time measurement (Zühlke 2012)

Trend from spot reading measurement is generated by comparing periodically determined short-time measurements. This also makes it possible to declare a statement, regarding defined limit values, that are considered "safe". Observations of changes in the condition of the insulation material over measured periods of time are thus transferable.

Dielectric Absorption Ratio (DAR) describes the behaviour when the capacitive charging current converges relatively quickly to zero after applying the test voltage. However, it takes some time (possibly hours) before the molecules of the dielectric are polarized and the absorption current drops. In addition, a leakage current occurs which superimposes the change in the charging currents. Over time, the curve of insulation resistance increases and flattens out. The dielectric absorption ratio can be represented by point measurements at fixed times and the derived ratio. The comparison times are usually 30 and 60 seconds. As a result, the measured values are significantly less affected by external influences, such as temperature changes, than point measurements at intervals of several hours.

$$DAR = \frac{R_{after\ 30\ seconds}}{R_{after\ 60\ seconds}} \hspace{2cm} (2\text{-}1)$$

Polarization Index (PI): in order to describe the condition of the insulation material, the "mobility of the molecules" must be described more precisely. For this purpose, the time interval between the comparison points must be increased. For the polarization index, the measured value after one minute is set in proportion to the value after ten minutes. By applying a direct current for a longer time, the molecules are collectively aligned and the polarization index, as an extended method of DAR testing, represents the polarization with a proportional value.

$$PI = \frac{R_{10\ min}}{R_{1\ min}} \hspace{2cm} (2\text{-}2)$$

Dielectric Discharge Test (DD-Test): with this test procedure the test current is switched off. After the capacitive discharge of the insulator, the so-called dielectric reabsorption current can be measured. This is the counterpart to the dielectric absorption current generated when the test voltage is applied. For this purpose, the insulator is charged sufficiently long until the insulation resistance stabilizes. Then the insulator is discharged via a resistor in the megohmmeter and the current is measured.

$$DD = \frac{I_{1\,min}}{U_{test} \cdot C} \qquad \text{(2-3)}$$

A test sequence of different test methods provides different information about the condition of the insulating material. This primarily requires knowledge of the components of the test current.
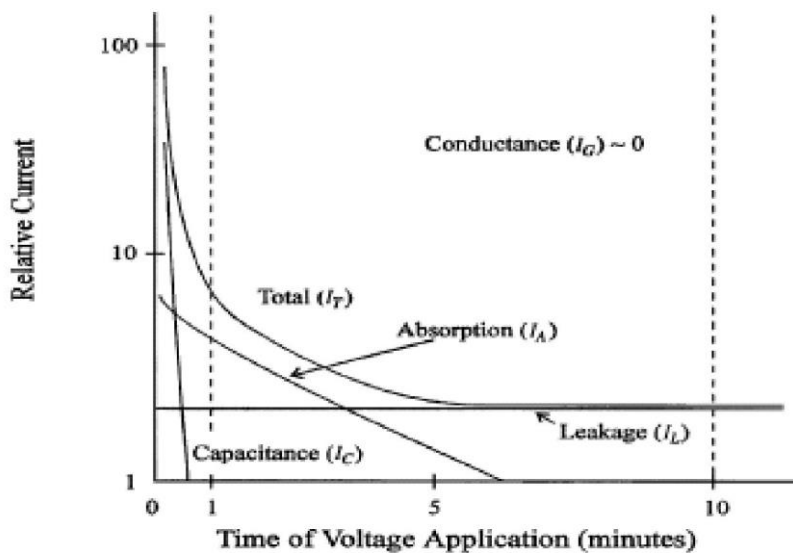
Figure 4: Components of current (IEEE 43, p. 8)

The Institute of Electrical and Electronics Engineers (IEEE 43, pp. 3–9) defines the four currents as follows.

The **conduction current** (I_G) is constant in time. It passes through the bulk insulation from the grounded surface to the high-voltage conductor that depends on the type of insulation system.

Also constant with time is the **leakage current** (I_L) This usually exists between exposed conductors. The magnitude of the leakage current is dependent upon temperature and the amount of conductive material, e.g. moisture or contamination on the surface of the insulation.

A current of relatively high magnitude and short duration is the **geometric capacitive current** (I_C). Furthermore, it decays exponentially with time of voltage application and is reversible. It depends on the internal resistance of the measuring instrument and the geometric capacitance.

The **absorption current** (I_A) results from molecular polarizing and electron drift, which both decay over period of time of voltage application at a decreasing rate from a comparatively high initial value to nearly zero. Accordingly, the absorption discharge current depends on the type and condition of the insulation system. It will decay from a high initial value to nearly zero with the same characteristics as the initial charging current but with the opposite polarity. This decay may take more than 30 minutes depending on the insulation type and size of the test specimen.

The different currents provide a comprehensive picture with sufficient parameters to assess the overall condition of the material.

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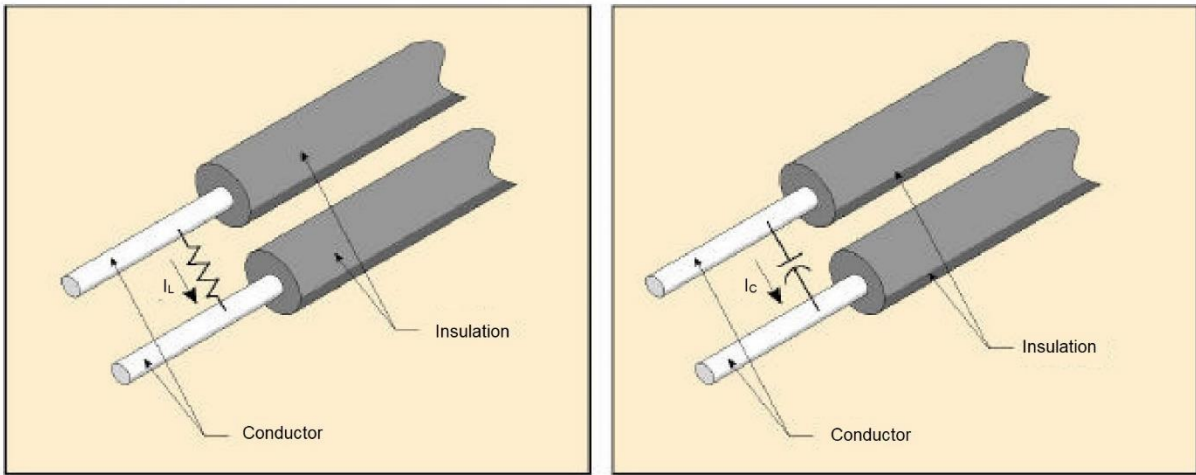
Figure 5 leakage current (I$_L$) and geometric capacitive current (I$_C$) (Fluke)

If two or more conductors are laid in a cable duct, they behave like a capacitor. Due to this capacitive effect, a leakage current flows through the conductor insulation. This current lasts only a few seconds when the DC voltage is supplied and converges towards zero when the insulation is fully charged to the test voltage. In low capacitive devices, the capacitive current is higher than the leakage current, but normally disappears when data recording begins. For this reason, it is important to wait until the measured value has "settled". In contrast, when measuring high-capacitance elements, the leakage current caused by the capacitive charging can last a very long time until a "steady mode" has taken place.

2.2.1 Three terminal test (guard-terminal)

When measuring the insulation resistance of insulation resistances with more than 100 MΩ, the measurements can be falsified by surface leakage currents. These flow off through the moisture or dirt deposits on the surface of the insulator. A resistor is created parallel to the actual insulation resistance (see Figure 6). And since surface leakage currents reach much lower values than the leakage currents, the measured resistance decreases. To exclude these surface leakage currents from the measurement, some measuring instruments have a third, so-called guard connection. The surface leakage current flowing on the surface of the insulator is picked up via this guard connection and excluded from the measurement.
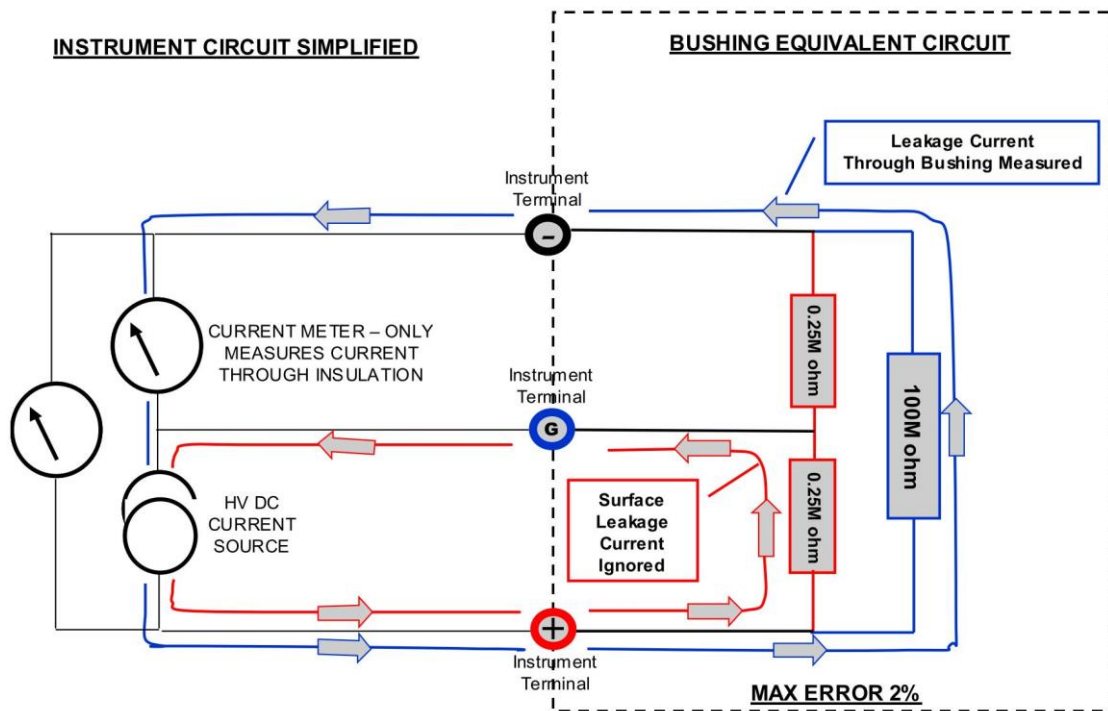
Figure 6 Measurement of a bushing insulator with exclusion of the surface leakage current -schematic circuit diagram - (Megger, p. 3)

As one can see in Figure 6 the circuit represents a surface leakage current in red, parallel with the real leakage current. Without the guard connected the instrument will measure then a total resistance of about 0.49 MΩ instead of real 100 MΩ. By using the guard terminal, the surface leakage current can be excluded from the measurement.

„The guard terminal must be connected to a surface that allows surface currents to flow, which is not the case of insulants such as cable or transformer insulation materials. Thorough knowledge of the possible paths taken by the test current when flowing through the element tested is crucial for choosing where to position the connection to the guard terminal." (Chauvin Arnoux, p. 7).

2.2.2 Influence of temperature

In the literature (Ose 2013, p. 20; Matula 1979) real resistances are temperature-dependent and the change is described by temperature coefficients. In most insulation resistance measurements, the range of temperature differences is in a relatively small interval. At limits within which the material properties change little, a linear change in the temperature difference is assumed.

$$R_T = R_{T\_0} \cdot \alpha[1 + (T - T_0)] \tag{2-4}$$

with

R_T	Resistance at temperature T
R_{T_0}	Resistance at reference temperature
α	Correction of insulation resistance

For materials with non neglectable changes in material properties within the temperature interval, the resistance value vary quasi exponentially.

$$R_T = R_{T\_0} \cdot e^{-b\left(\frac{1}{T_0} - \frac{1}{T}\right)} \tag{2-5}$$

with

b   Correction of insulation resistance

Measurement should be carried out in similar temperature conditions. If it is not possible the results can be compared by conversion to a reference temperature. However, as other factors have a direct effect on the measurement result, a standardised measurement method is essential in order to achieve the best possible comparability.

2.3 Standards and Recommendations

"A standard is a document, established by consensus and approved by a recognized body, that provides, for common and repeated use, rules, guidelines or characteristics for activities or their results, aimed at the achievement of the optimum degree of order in a given context." (Incore cables, p. 2)

Standardisation is a regulated process and each country has its own policy in developing standards. A standard is generally initiated by interest groups to establish guidelines for the requirements for goods and services and to define rules for testing. Certification and accreditation of the goods or service is created. This contributes to the development of suitable and safe products, production processes and services. For the engineer, the standard provides detailed descriptions in the form of EU directives, national laws and regulations.

In the case of application, various regulations can come into play. Both offshore regulations of a classification society and national law are binding. For example, DNV-GL (Recommended Practice DNV-RP-J301, p. 86) recommends random sampling test of components and the entire cable to monitor the quality of the cable. DNV-GL also complies with the requirements for sample testing laid down in IEC standards and CIGRÉ guidelines.
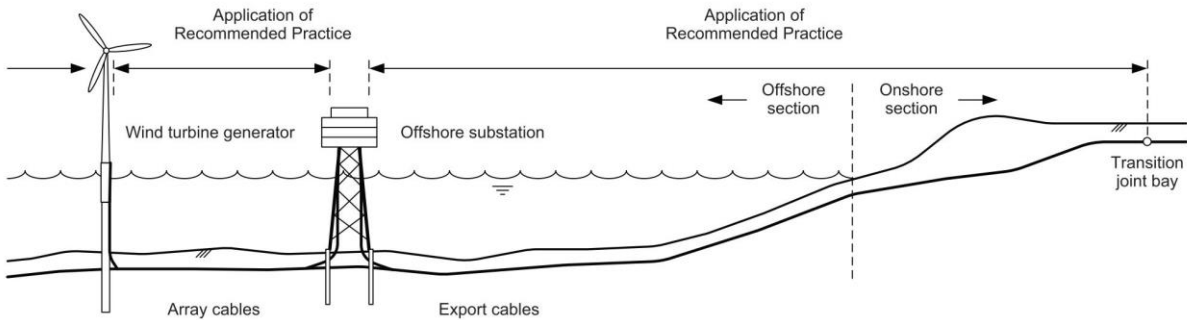
Figure 7: Applicable Areas of DNV-RP-J301 (Recommended Practice DNV-RP-J301, p. 9)

There are various responsibilities and binding recommendations drawn up by different classification societies. In Europe, cable properties are most frequently tested according to the standards of the

International Electronical Commission, or IEC standards for short. The IEC is an international standards organisation based in Geneva, which drafts IEC standards in the fields of electrical engineering and electronics. Although national standards organizations such as ANSI (American National Standards Institute), DIN (German Institute for Standardization), or NEK (Norsk Elektroteknisk Komitee) are members of the IEC, there are still differences between the national standards.

standards.html

Figure 8 interactive map, please double click
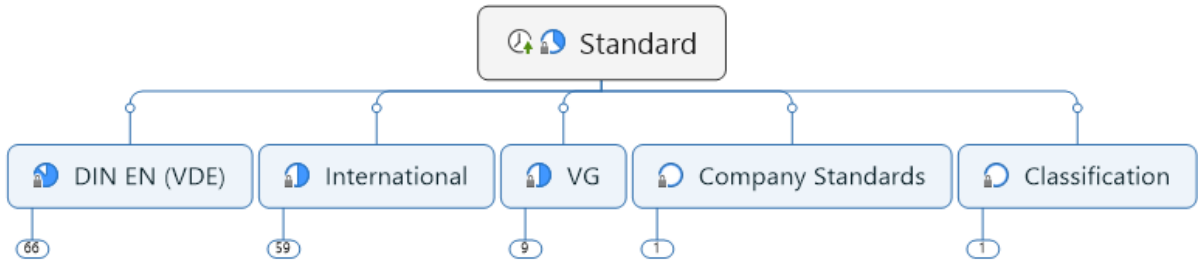
Figure 9 Principal arrangement of standards

Cable manufacturers offer different certificates, as cables on land are subject to national regulations that may prescribe different test methods. „Test procedures are recognised as simple and fundamental. […] They have also been widely recognized and have also been used for other cables., [...], communications cables and control lines, including ship cables and cables used for offshore applications" (DIN EN 60811-100, p. 4). See figure BLA for the complex crosslinking. In practice, however, this transferability takes place only to a limited extent.

In 2009, NEK decided to issue the NEK 606 as a technical specification and the name of the standard was changed to NEK TS 606. This decision was taken to avoid the specification being interpreted as an international standard and not as a technical specification. As the specification is not published by the IEC, it cannot be interpreted as a national standard. Companies have their own specifications and often use the NEK TS 606 as a guide for cable specification. It is easier to refer to NEK TS 606 than to create your own specification. They can also assume that they agree with other companies that also refer to NEK TS 606.

In the context of this work it was examined which regulations, specifications and recommendations already exist for low voltage cables for measuring the insulation resistance in connection with water and to what extent there are overlaps. The demand is shown in detail using the example of the water used.

Table 1 water specifications used in standards

Designation	Water	Temperature

ASTM D 3032	water contains: 5 % NaCl and 5-10 % wetting agent	23 °C ± 5 °C
DIN EN ISO 13628	potable water; later town-mains water	no limitations
IEC 60092-350	not specifications	50 °C ± 2 °C
IEEE 43	<0.25µS/cm	no limitations
VDE 0276-605	tap water, if not specified otherwise for mechanical testing deionized water	no limitations
VDE 0304; DIN EN 60216	no specifications	no limitations
VDE 0472-501	no specifications	reference to type specific test document
VDE 073-811-402; DIN EN 60811-402	boiled or deionized water	< 90 °C
VDE 0481-395; DIN 50395	solution of sodium chloride	< 90 °C
VG 95218-29	no specifications	23 °C ± 3 °C
IEEE 1580	no specifications	10 °C bis 75 °C

Most standards do not define water in more detail. With regard to temperature, reference is made to other standards or no explicit restriction is made. The temperature used must be explicitly mentioned in all test reports. However, the composition of the water is neglected. In the regulations presented in Table 1 water is used almost exclusively as the electrolyte for insulation resistance measurements. If cables are artificially aged, then almost exclusively in the heating cabinet in air or other gas mixtures. In addition, the temperature at which the water is set up for testing is different or unspecified. In conclusion, it can be said that the reference temperature of the insulation test is different among the reviewed standards.

The following sub-chapters contain regulations, specifications and recommendations which refer to the test method of insulation resistance measurement through concrete specifications and/or formulations. Documents from classification societies are presented in the "Guidelines" section, as they are not binding as a substitute for national standards. As a common feature all presented documents use the test method of insulation resistance. Standards are defined as either normative or informative. Normative regulations are indispensable for their application. Informative regulations provide additional information for understanding or to limit the further use of the measured values. The temperature increase during insulation resistance measurements in connection with artificial ageing plays an additional role here.

2.3.1 Standards – Normative

These are the normative standards:

ASTM D 1141-98	Standard practice of the Preparation of Substitute Ocean Water
ASTM D 257	Test Methods for DC Resistance or Conductance of Insulating Materials
ASTM D 3032	Standard Test Methods for Hookup Wire Insulation
DIN EN 50289-1-4	Spezifikationen für Prüfverfahren
DIN EN 60811-408	Kabel, isolierte Leitungen und Glasfaserkabel - Prüfverfahren für nichtmetallene Werkstoffe - Teil 408: Sonstige Prüfungen - Langzeit(Lebensdauer)- Prüfung für Polyethylen- und Polypropylenmischungen
DIN EN ISO 13628-5	Petroleum and natural gas industries – Design and operation of subsea production systems
VDE 0276-605	Starkstromkabel — Ergänzende Prüfverfahren
VDE 0472-502	Prüfung an Kabeln und isolierten Leitungen - Isolationswiderstand und spezifischer Durchgangswiderstand
VDE 0472-509	Prüfung an Kabeln und isolierten Leitungen - Spannungsfestigkeit von Kabeln, Leitungen und Schnüren für Fernmelde- und Informationsverarbeitungsanlagen
VDE 0481-395; DIN EN 50395	Elektrische Prüfverfahren für Niederspannungskabel und -leitungen
IEC 60092-350	Electric installations in ships - General construction and test methods of power, control and instrumentation cables for shipboard and offshore applications
VG 95218-2	Kabel und Leitungen — Fachgrundnorm
VG 95218-29	Kabel und Leitungen —Kabel, längswasserdicht, querwasserdicht, Bauartnorm

In Figure 10 **Fehler! Verweisquelle konnte nicht gefunden werden.**the normative standards are highlighted in green and all the other standards and regulations examined are greyed out. See BLA as an interactive map, all the standards and regulations tagged with a green flag are normative.

It is obvious that the rules refer to each other. Some standards act as a hub and have become relevant. The only exception is ASTM D 1141 (ASTM D 1141-98). This American standard defines the composition of artificial seawater. Although the appendix to DIN 50905-4 also contains (DIN 50905-4) a guide for the production of artificial seawater, it does not indicate a temperature and is therefore less precise with regard to the pH value. It should also be noted that some standards have been withdrawn. However, reference is made to these with the note that the relevant information is still valid. DIN EN 50395 contains electrical test procedures for the

Figure 10 Overview with active filter (normative standards only)

testing of harmonized low-voltage cables and lines, in particular those with nominal voltages up to 450/750 V. For insulation condition measurements at maximum permissible conductor temperatures of 90°C (DIN EN 50395, pp. 8–9) the test set-up is described down below:

- There is a DC voltage between 80 V and 500 V between the conductor and the water;
- Insulation resistance of each core must be measured 1 minute after applying the voltage. This value is used to calculate the insulation resistance of a 1 km length for each core;
- The length of the sample is 5 m;
- The sample ends protrude about 250 mm out of the water;
- The test temperature is 20° C ± 15° C.

The range of test voltage for direct current is defined in all standards in a similar wording: "The test shall be carried out with a DC voltage between 80 V and 500 V for a minimum duration of one minute, unless otherwise specified in the corresponding cable specification." (DIN EN 50289-1-4, p. 5). There is a common definition with regard to the measurement duration. One minute after applying the voltage, the insulation resistance is measured.

The specifications for the length of the specimen vary, a range from 1 m to 10 m is recommended. A range from 1 m to 10 m is indicated. The length of the ends protruding from the water varies minimally

between 0.2 m and 0.3 m. The following expression is representative: "In the standards, [...] if no other length is specified, the length of the specimen in the water bath is approximately 5 m. It is placed in the water bath in such a way that the ends protrude by 0.2 m. The specimen is then bent into a ring with a diameter of about 15 * d, but at least 0.20 m (d = nominal diameter of the insulating sheath)." (DIN VDE 0472-502, pp. 2–3).

There are hardly any regulations for the arrangement of the cables in the water bath. For example, the boundary conditions in DIN EN ISO 13628-5 are formulated very precisely, but the position of the samples is missing.

"The individual insulated conductors shall be fully immersed in a tank filled with potable water. Insulation resistance shall be measured. The specimens shall then be subjected to a minimum hydrostatic pressure of 3.5 MPa (500 psi), or maximum hydrostatic pressure at service water depth, whichever is greater, for a minimum period of 22 h, and then insulation-resistance tested while still under pressure. The value of the insulation resistance shall not be less than the value defined in 7.2.6.2. [500M ohm km at 500 V DC]" (DIN EN ISO 13628-5, p. 42).

To collect information about the arrangement, retrieval of standards related to artificial ageing and insulation resistance measurements at elevated temperatures, was carried out. Therefore, standards related to artificial ageing and insulation resistance measurements are used for cable positioning. A limiting factor here is that artificial ageing is carried out in other media than water in these standards.

According to DIN EN 60811-401 (DIN EN 60811-401, p. 7) the test specimens must be 20 mm away from the adjacent test specimens and must also hang centrally. In addition, the test specimens must not take up more than 2 % of the oven volume. In addition, the positioning in DIN VDE 0472-502 is further specified: "The distance between the sample and the walls of the heating cabinet should be at least 5 cm". (DIN VDE 0472-502, S. 3).

Furthermore, when testing the direct voltage resistance of the insulation (DIN EN 50395, p. 9) a measurement is set-up in which the medium is specified as a solution of sodium chloride (10 g/l). This standard also lists the pin assignments. "Connect the negative pole of a 220 V DC source to the conductor of the sample and the positive pole to a copper electrode immersed in the solution. The test is considered passed if it does not produce a disruptive discharge." This means that this check is not used to obtain numerical data. When testing for capacity change and insulation resistance after water storage in the VG 95218-2 (VG 95218-2, p. 98) the measured values are transformed on the basis of proportional values. All the measured values are set in relation to the initial value and the resulting ratio is published.

In ASTM D3032 the composition of water is defined as "containing 0.05 to 0.10 % wetting agent (Triton X-100 by *Rohm & Haas Co.*) "and the temperature given with: "water bath at 23°C +-5°C". Further, the vessel has to be insulated and large enough to immerse the specimen. The specimens are well described: "the specimen shall consist of 8.3 m (26 ft), remove insulation for 25 mm (1 inch) at each end and twist the ends together, immerse 8 m". The test programme sequences are described in full detail. It starts with an initial resistance measurement "for non-typical values, discard specimen with gross defect (insulation resistance less than 1×10^6 Ω between the conductor and the water bath)". At this point non-typical values are defined. But the position and the distance of the electrode in water is missing. The programme continues with the second measurement "after 4 hours remeasure at 500 (+-10%) d.c V, after electrification time of 1 minute". The values will be converted to the standard unit,

despite the length being a non SI-unit: "calculate as ohm-1000 ft --> (R x L) / 10000, with L immersed length of 25 ft and R measured resistance in ohm" (ASTM D 3032, p. 3). The instruction contains all the necessary information, except the position of the cable and the electrode in the vessel. According to the objective of simulating realistic test conditions, artificial seawater is chosen as the inlay medium. Mixture is to be carried out in accordance with ASTM D 1141 (ASTM D 1141-98). The ready-mixed artificial seawater is filtered off after one day standing and adjusted to pH 7-8 with sodium hydroxide solution (DIN 50905-4).

"The test equipment must have a suitable minimum range, typically 5×10^5 Megaohm and must comply with the voltage specified in the corresponding specification"(DIN EN 50289-1-4, p. 5).

In addition, DIN VDE 0472-502 (DIN VDE 0472-502, p. 2) requires an error limit G = (10 + 0.0002 * W) % of the nominal value in addition to the measuring range of at least 2×10^5 MΩ. For W, the numerical value of the insulation resistance in megohm must not exceed 2×10^5.

The minimum value for the insulation resistance is calculated using the following formula:

$$R = 0.0367 \cdot \rho \cdot 10^{-8} \cdot \log\left(\frac{D}{d}\right)$$

(2-6)

with

R   Insulation resistance in MΩ · km
ρ   specific resistance in Ω · m
d   diameter of the conductor
D   d + twice the required mean of the sheath

For the evaluation of long-term characteristic values, a limit value of 50 % relative to the initial value is often used (DIN EN 60216-2, p. 5).

The time at which the initial value is to be measured can be interpreted. According to IEC 60092-350 (IEC 60092-350, p. 22) the first measurement should be made 1 hour after immersion. The pre-treatment is taken into account according to DIN VDE 0472-502: "The measurement starts at the very latest 5 minutes after the end of the pre-treatment. " (DIN VDE 0472-502, p. 2). The time of immersion in the water bath is defined as pre-treatment in DIN EN 60216-1: "For determining the initial value of the property [...]. these specimens shall be pre-treated by storage for two days (i.e. for 48 hours +/- 6 ours) at the lowest stage of the storage temperature intended for the test [...]. in order to achieve a stable value" (DIN-EN 60216-1, p. 13). Then the initial value is to be measured. This means that further processing of the data is refined, for example in accordance with DIN VDE 0276-605 ("Test report with diagrams showing capacity increases expressed as a percentage of the first day value". (DIN VDE 0276-605, p. 174). And on the other hand, the reference temperature is then always the temperature of the first measurement.

The procedure described in IEC 60092-350 is giving additional information about other measurements. "Measure the insulation resistance at ambient temperature with a DC voltage of 80 V to 500 V. An AC voltage test may be carried out beforehand, but not a DC voltage test. The measurement usually takes place 1 minute after the voltage is applied. In order to achieve an essential stationary state, however, the measurement can be extended to a maximum of 5 minutes in certain cases. The cable to be tested

is connected to the test equipment as follows: Core against core, core against metal jacket/shield" (DIN VDE 0472-509, p. 2), or "inner conductor to outer conductor, outer conductor earthed", (DIN EN 50289-1-4, p. 5) and screen against water. The minimum distance between two measurements is set at 4 hours in ASTM D 3032 (ASTM D 3032, p. 3).

In general, the measured values have to be converted to the reference unit MΩ · km as described for example in standard DIN EN 50289-1-4 (DIN EN 50289-1-4, pp. 5–6):

$$R_i = \frac{R_m}{1000} \cdot L$$ (2-7)

with
R_i Insulation resistance in MΩ · km
R_m measured resistance of the specimen, in MΩ
L   Length of the specimen, in m

The value of the insulation resistance between the core and the water bath shall not fall below the defined value 500 MΩ · km at 500 V DC in accordance with (DIN EN ISO 13628-5, p. 39)

- 1 MΩ · km at 200 V in accordance with (VG 95218-29, pp. 44–46)

2.3.2 Standards – Informative

These are the informative standards:

DIN EN 50363-10-2	Isolier-, Mantel- und Umhüllungswerkstoffe für Niederspannungskabel und -leitungen - Diverse Mantelmischungen - Thermoplastisches Polyurethan
DIN EN 50905-4	Korrosion der Metalle - Durchführung von chemischen Korrosionsversuchen ohne mechanische Belastung in Flüssigkeiten im Laboratorium
DIN EN 5290-2-20	Kommunikationskabel - Gemeinsame Regeln für Entwicklung und Konstruktion – Allgemeines
DIN EN 60216-1	Elektroisolierstoffe - Eigenschaften hinsichtlich des thermischen Langzeitverhaltens Warmlagerungsverfahren und Auswertung von Prüfergebnissen
DIN EN 60216-2	Leitfaden zur Bestimmung thermischer Langzeiteigenschaften von Elektroisolierstoffen - Auswahl der Prüfmerkmale
DIN EN 60216-3	Anweisungen zur Berechnung thermischer Langzeitkennwerte
DIN EN 60216-5	Bestimmung des relativen thermischen Lebensdauer-Indexes (RTE) von Elektroisolierstoffen
DIN EN 60216-6	Bestimmung der Thermischen Langzeitkennwerte (TI und RTE) eines Isolierstoffes unter Anwendung des Festzeitrahmenverfahrens
DIN EN 60216-7-1	Beschleunigte Ermittlung der relativen thermischen Beständigkeit mit analytischen Prüfverfahren (RTE_A) - Anleitung für die Berechnung auf der Grundlage der Aktivierungsenergie
DIN EN 60216-8	Anweisungen zur Berechnung von charakteristischen Werten zum thermischen Langzeitverhalten unter Verwendung vereinfachter Verfahren
DIN EN 60228	Leiter für Kabel und isolierte Leitungen
DIN EN 60811-100	Kabel, isolierte Leitungen und Glasfaserkabel - Prüfverfahren für nichtmetallene Werkstoffe - Allgemeines
DIN EN 60811-301	Elektrische Prüfungen - Messung der Dielektrizitätskonstanten von Füllmassen bei 23°C
DIN EN 60811-401	Sonstige Prüfungen - Thermische Alterungsverfahren - Alterung im Wärmeschrank
DIN EN 60811-402	Prüfverfahren für nichtmetallene Werkstoffe - Sonstige Prüfungen - Wasseraufnahmeprüfung
DIN EN 60811-606	Prüfverfahren für nichtmetallene Werkstoffe - Teil 606 Physikalische Verfahren zur Bestimmung der Dichte
DIN EN 61400-3	Windenergieanlagen - Auslegungsanforderungen für Windenergieanlagen auf offener See
DIN EN ISO 13628-6	Petroleum and natural gas industries – Design and operation of subsea production systems - Part 6: Subsea production control systems
VDE 0207-23	Isolier- und Mantelmischungen für Kabel und isolierte Leitungen
VDE 0281-2	Cables of rated voltages up to and including 450/750 V and having thermoplastic insulation

In DIN EN 61400-3, the use of seawater is combined with various applications, according to which seawater is also described in more detail. In marine environment oxidation depends on the presence

of a conductive ionic electrolyte which is provided by seawater. According to the conclusions, key values of seawater are (DIN EN 61400-3, p. 117):

- Type and mass of dissolved salts and pollutants
- Dissolved oxygen
- Temperature
- Motion and flow

In addition, UV radiation and vegetation are named as further influencing factors. "The upper flood zone and the lower part of the splash zone are usually affected by vegetation." (DIN EN 61400-3, p. 118).

According to DIN EN ISO 13628-5 (DIN EN ISO 13628-5, p. 39), the sheath of an electrical cable in a marine environment must be made of a polymeric material with protection against UV radiation and oxidation and must comply with the manufacturer's written specification. The selected material should be extruded continuously and concentrically over the cores to obtain a uniform cross-section. The material must be compatible with seawater and the specified operating fluids used during manufacture, installation and maintenance and must not impair the quality of other materials with which it may come into contact. For seawater resistance the samples shall be tested according to VG 95218-29 (VG 95218-29, p. 48). Thus testing is performed by winding specimens (each 1 m or 10 m long) around a stainless steel mandrel, the diameter of which is 5 times the cable diameter. There should be three full turns next to each other on the mandrel and the ends should protrude from the water. The applied test voltage (DC) should be between 80 V - 200 V. At a test temperature of 70 °C ± 2 °C, the sample is placed in water for a test period of up to 28 days. The samples are then cooled in water to a temperature of 23 °C ± 2 °C within 24 hours. The insulation resistance is measured within the last 30 minutes of the cooling process:

- between 2 adjacent conductors;
- between the screen and a directly adjacent conductor;
- between one conductor and the water.

The insulation resistance should be at least 1 MΩ · km. With regard to the duration of the test, dependent on the wall thickness, DIN EN 60811-402 (DIN EN 60811-402, pp. 6–8) requires storage to be in boiled or deionised water. Length of time:

- up to 1 mm 14 days
- 1 - 1.5 mm 21 days
- 1.5 mm 28 days

A further note regarding the exchange of the storage medium can be found in DIN EN 60811-409 (DIN EN 60811-409, p. 7). "In case of ageing in the heating cabinet, at least 8 and a maximum of 20 complete air changes should be carried out per hour."

Materials currently used in metal cables and fiber optic cables are described below. Other materials are not prohibited, but professional advice is recommended.

EN 50290-2-X Abbreviation	EN ISO 11469 Abbreviation	Material
PVC	PVC	Polyvinylchlorid
LLDPE, LDPE, MDPE, HDPE	PE-LLD, -LD, -MD, -HD	Polyethylen
PP	PP	Polypropylen
PA	PA-6, -12, -66	Polyamid (Nylon)
SiR	FMQ	Silicon-Elastomer (Gummi)
PU	PU	Polyurethan
PBT	PBT	Poly(butylen terephthalat)
TPE	TPA, TPC, TPO, TPS, TPU, TPV, TPZ	Thermoplastisches Elastomer
FEP	E/PF	Fluoriniertes Ethylen-Propylen
HFFR-LS		Mischung auf Polyolefin-Basis mit Flammschutzmittel als Additiv
XLPE	PE-X	PE – vernetzt oder vernetzbar

Figure 11 Nomenclature of polymers (DIN EN 50290-2-20, p. 5)

It is mandatory in DIN EN 60811-412 (DIN EN 60811-412, p. 5) to conduct no tests within 16 hours after extrusion or cross-linking of the insulation or sheath compounds. It is also pointed out that samples of each colour must always be taken (DIN EN 60811-408). Coloured of polymers are produced through the addition of additives which can influence the test.

"The maximum operating temperature is based on thermal degradation, heat deformation properties and the thermal sensitivity of the dielectric properties. [...] Unless otherwise specified, all materials described in EN 50290-2 are suitable for 70° C operating temperature" (DIN EN 50290-2-20, p. 5).

The glass transition temperature, also known as the dropping point determines the storage temperature. Temperature for ageing in a convection oven for seven days at 60+2 °C or at 70+2 °C depends on the fillings with a required dropping point above the storage temperature. (DIN EN 60811-408, p. 6).

The measured values of the insulation resistance must be corrected to the reference temperature of 20 °C by using a suitable temperature correction factor. The suitable temperature correction factor is determined by experimental results of the insulation material concerned (IEC 60092-350, p. 22).

According to DIN VDE 0472-502 the conversion is done according to:

$$R_0 = R_t \cdot e^{\alpha(t - t_0)} \tag{2-8}$$

with

R_t	Resistance in Ω at temperature
R_0	Resistance in Ω at reference temperature
α	Correction of insulation resistance

An aggravating factor is the fact, that the sum of material properties of artificially aged specimen do not deteriorate all at the same rate during warm storage. This means that on the one hand the temperature correction coefficient can change and on the other hand there are different limit values for different applications. This leads to the assumption that more than one temperature index can be attributed to a material. (DIN-EN 60216-1, p. 5).

The IEC 60216-5 standard (DIN EN 60216-5) describes artificial ageing in air to characterise long-term behaviour. Long-term characteristic values for each material are determined using different properties or limit values. Limit values serve as an indicator for the determination of the degree of thermal degradation.

"The limit can be defined as:
- percentage increase or decrease of the measured characteristic value, relative to the initial value. [...]
- fixed characteristic value. This value can be selected to meet normal operating requirements. [...]

The limit value should be selected so that it indicates the degree of thermal degradation of the insulating material, [...]" (DIN EN 60216-6, p. 13).

For the graphical determination of the temperature index, the x-coordinate, which is defined by the average of the reciprocal values of the thermodynamic (absolute) temperature, at which the limit value is reached and the y-coordinate is the logarithm of the ageing time, are plotted on a diagram." (DIN EN 60216-6, p. 14). It is assumed that the relationship between absolute temperature and ageing time is linear. The extrapolation of duration for degradation is based on the theoretical 1st order chemical reaction, with small deviations from the ideal Arrhenius behaviour. Therefore, the validity of the underlying physicochemical model must be checked. A confidence limit is formed for the temperature index[3]. In addition, the halving interval[4] is introduced for better representation of the rate of changes in time to failure with temperature (DIN-EN 60216-1, p. 5). The thermal long-term characteristic values are summarized:

- Temperature index (TI)
- Halving interval (HIC).

In accordance with DIN 60216-3 (DIN-EN 60216-3, p. 11), the following statistical conditions must be met for determination using the mathematical method:

- Fischer Test for linearity
- Equality of the variances (Bartletts Xi^2 Test)

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[3] Temperature index (TI): numerical value of the temperature in degrees Celsius derived from the long-term thematic relationship for a time of 20 000h (or another fixed time) DIN-EN 60216-1, p. 7.

[4] Halving interval (HIC): numerical value of the temperature difference in Kelvin, which halves the time to failure at the temperature of the TI. ibid.

- F-Test (ratio of the deviations of the regression line to the combined variance within the samples) to verify the validity of the Arrhenius relationship when applying the test results

In case of graphical derivation or if the statistical conditions are not met, the values must be marked with the footnote g. If the derivation is based on the simplified method according to IEC 60216-8, footnote s shall be used. Figure 12 shows the temperature index. After it has been defined, the regression line is drawn into the thermal long-term diagram, i.e. into a diagram with y = log(time to failure) as ordinate and x = temperature as abscissa. "The long-term thermal diagram can be completed by drawing in the curve of the lower 95 % confidence curve" (DIN-EN 60216-3, 21).
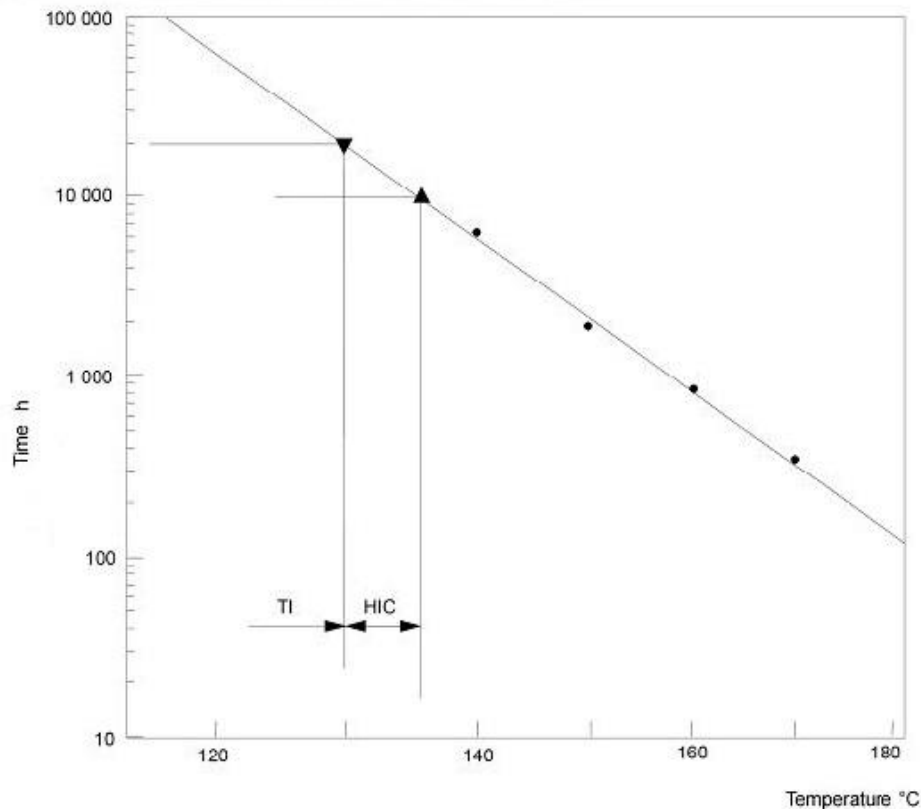

Figure 12 Long term diagram (DIN-EN 60216-1, p. 16)

"The times to failure in this test cannot be quantitatively related to the service life of insulating materials in actual use, but provide a relative indication of such service life under the specific parameters of the test." According to ASTM 3032 (ASTM D 3032, p. 10-14), the limitations to the parameters of the test must be made when using this method. It is worth mentioning, that any change of the test conditions changes the result and the effects of different parameters influence the measurement result differently, when ageing in a convection oven and measurements in a water bath. "Because experience has shown that different laboratory ovens rarely give duplicate results after thermal exposure test, it is strongly recommended that this method be limited to evaluation of the relative performance of two or more insulation systems tested at the same time. [...] Numerical limits of precision cannot be set at this time." (ibid.). It is strongly recommended to limit this method to the evaluation of the relative performance of two or more parameters of insulation systems, tested simultaneously. This means that numerical accuracy limits cannot be set at present.

In order to minimize changing test conditions , the test DIN 60811-402 (DIN EN 60811-402, pp. 6–8) works with ratio values. The measured values are recorded at defined points in time and compared with the reference or previous measured values and then processed further with the ratio.

"The long-term thermal behaviour of an electrical insulation material always applies only to a specific property and a specific limit value" (DIN-EN 60216-1, p. 17). As different properties change at different rates, a material may have different temperature indices, as already mentioned. Knowing that the condition of the underlying physicochemical model is valid for interfacial transition "gas-gas" leads to a systematic error. The statement "The deviations of the values for x from the linear relationship are normally distributed with a variance that is independent of the ageing time" (DIN EN 60216-6, pp. 14–15), must be interpreted as an assumption.

2.3.3   Guidelines

At this point, additional information from DNV-GL recommendations and class regulations are sorted thematically. Repetitions should be avoided and not all statements of all recommendations need to be reproduced:

IEEE 43	Recommended Practice for Testing Insulation Resistance of Electric Machinery
IEEE 98	Standard for the Preparation of Test Procedures for the Thermal Evaluation of Solid
IEEE 1580	Recommended Practice for Marine Cable for Use on Shipboard and Fixed or Floating Facilities
DNV-RP-J301	Subsea Power Cables in Shallow Water Renewable Energy Applications
ITU-T G.976	Test methods applicable to optical fibre submarine cable systems
NEK 606	Cables for offshore installations - halogen-free and/or mud resistant -- Technical specification
TB 493	Non-destructive water-tree detection in XLPE cable insulation

According to recommended practice IEEE 43 (IEEE 43, p. 14) water should have a conductivity of not more than 0.25 µS/cm. It is therefore recommended to use deionized water to prevent chemical reactions due to the contents of the water. In relation to the selection of artificial seawater, the chemical activity should be determined.

The insulation resistance value varies exponentially with temperature. IEEE 43 (IEEE 43, p. 10) also points out the difference between the temperature dependence of the resistivity in metals and non-metallic materials, especially in good insulators. In metals, a higher temperature leads to a thermally excited movement that reduces the mean free path of electron movement and thus increases electron mobility and resistivity. In the case of insulators, on the other hand, an increase in temperature

supplies heat energy. This releases additional charge carriers and reduces the specific resistance. This temperature change affects all components of the measured current, with the exception of the capacitive current.
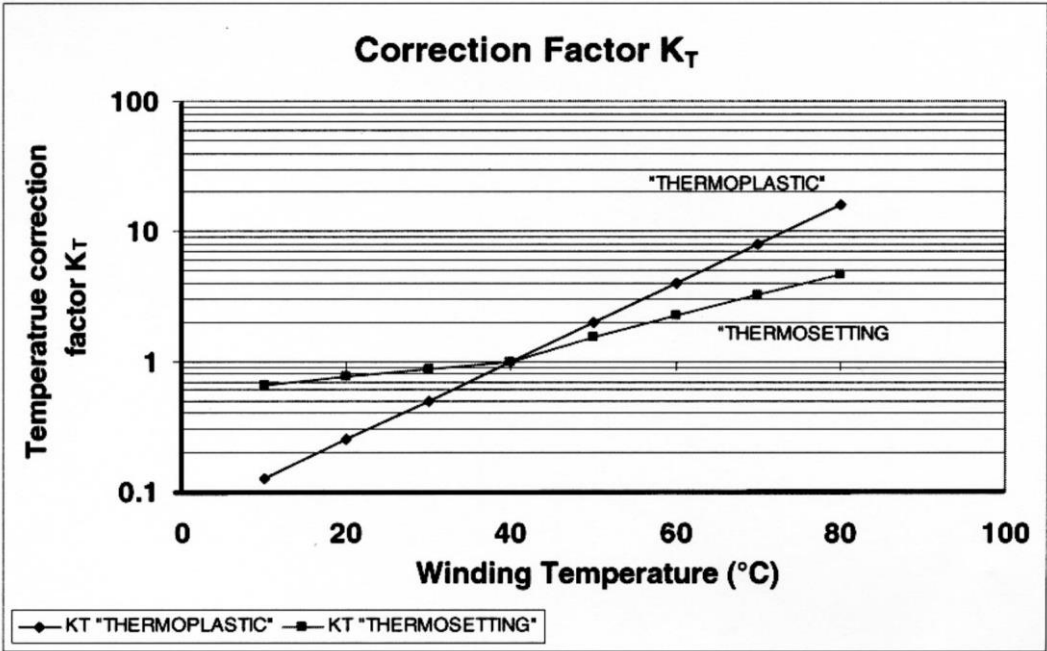

Figure 13 Correction of insulation resistance(K_T) for insulation systems (IEEE 43, p. 11)

Figure 13 shows temperature correction factors for insulators called "thermoplastic" (asphalt) and "thermosetting" (epoxy or polyester).

$$R_C = K_T \cdot R_T \tag{2-9}$$

with

R_C	Insulation resistance (in megohms) corrected to 40 °C
K_T	Insulation resistance temperature coefficient at temperature T °C
R_T	Insulation resistance (in megohms) at temperature T °C.

Recommended practice IEEE 1580 (IEEE 1580, p. 20) presents an adaptation of the method to determine insulation resistance as a function of temperature, which is summarised here. To determine the temperature correction factor, two samples with defined dimensions in length (60 m) and thickness of the insulation (1.14 mm) are immersed in a water bath. The temperature of the water bath is pre-selected, so that stable insulation resistance values are measured in the calibrated area of the measuring instrument at the lowest water bath temperature. The water bath should be equipped with heating, cooling and circulation systems. The ends of the samples must be at least 0.6 m above the water surface to avoid surface leakage currents. Before setting the water temperature to 10.0 °C, the samples should remain in the water for 16 hours at room temperature.

The DC resistance of the metal conductor should be measured at suitable intervals until the temperature remains constant for at least five minutes. The insulator can then be viewed at the same temperature as the water. Each of the two specimens shall be subjected to successive increasing water temperatures of 10.0 °C, 16.1 °C, 22.2 °C, 27.8 °C and 35.0 °C then decreasing again to 27.8 °C, 22.2 °C,

16.1 °C and 10.0 °C. The insulation resistance values shall be measured at each temperature after the equilibrium between insulator and water temperature has been established.

The measured values at a specific temperature are averaged for the two samples. These four mean values and the mean value of the individual values at 35.0 °C are applied to logarithmic paper. A continuous curve (usually a straight line) is to be drawn through the five points. The value of the insulation resistance at 15.6 °C, which is the normal temperature in IEEE 1580, can then be read from the graph.

The resistivity coefficient C for a temperature change of 1 °C shall be calculated to two positions after the decimal point. To do this, the insulation resistance read at 15.1 °C from the graph is divided by the insulation resistance at 16.1 °C. The temperature correction factor M required to correct to the standard test temperature of 15.6 °C is calculated from the following formula:

$$M = C^{(t-15.6)} \tag{2-10}$$

with

t   Actual test temperature in °C
C   Resistivity coefficient

This formula has been used to generate a table of temperature correction factors (**Fehler! Verweisquelle konnte nicht gefunden werden.**).

Table 2 Temperature correction factor M for adjusting insulation resistance to 15.6 °C (IEEE 1580, pp. 49–50)

Temp °C (°F)	Resistivity coefficient *C* (see Results)										
	1.02	1.04	1.06	1.08	1.10	1.12	1.14	1.16	1.18	1.20	1.22
5.0 (41.0)	0.81	0.66	0.54	0.44	0.36	0.30	0.25	0.21	0.17	0.14	0.12
6.0 (42.8)	0.83	0.69	0.57	0.48	0.40	0.34	0.28	0.24	0.20	0.17	0.15
7.0 (44.6)	0.84	0.71	0.61	0.52	0.44	0.38	0.32	0.28	0.24	0.21	0.18
8.0 (46.4)	0.86	0.74	0.64	0.56	0.48	0.42	0.37	0.32	0.28	0.25	0.22
9.0 (48.2)	0.88	0.77	0.68	0.60	0.53	0.47	0.42	0.38	0.34	0.30	0.27
10.0 (50.0)	0.90	0.80	0.72	0.65	0.59	0.53	0.48	0.44	0.40	0.36	0.33
11.0 (51.8)	0.91	0.83	0.76	0.70	0.65	0.59	0.55	0.51	0.47	0.43	0.40
12.0 (53.6)	0.93	0.87	0.81	0.76	0.71	0.66	0.62	0.59	0.55	0.52	0.49
13.0 (55.4)	0.95	0.90	0.86	0.82	0.78	0.74	0.71	0.68	0.65	0.62	0.60
14.0 (57.2)	0.97	0.94	0.91	0.88	0.86	0.83	0.81	0.79	0.77	0.75	0.73
15.0 (59.0)	0.99	0.98	0.97	0.95	0.94	0.93	0.92	0.91	0.91	0.90	0.89
15.6 (60.0)	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00

The same principles are used to perform the insulation resistance test in recommended practice IEEE 98 (IEEE 98, p. 9), but with the limitation that the correction factor is investigated for each test method to assess the thermal resistance of thermally aged solid electrical insulating materials (EI) in air. The insulation resistance should be measured after one minute with a direct current at an applied voltage of not less than 100 V nor more than 500 V. The conductor is connected to the negative pole. For water the calculation, according to IEEE 1580, is performed in a modified form. If the insulation resistance test is performed in water or air at a temperature other than 15.6 °C, multiply the measured value by the correction factor M shown in **Fehler! Verweisquelle konnte nicht gefunden werden.**. This results in the following formula for the insulation resistance *(IEEE 1580, p. 92):*

$$R = K\,M\,\log_{10}\left(\frac{D}{d}\right)$$

(2-11)

with

R	Insulation resistivity [MΩ · km]
K	Insulation resistance constant (from Table 12, Table 13, or Table 14) (MΩ·1000 ft)
M	Temperature correction factor to 15.6 °C
D	Diameter over the insulation
d	Diameter under the insulation

Besides the change of characteristics over time, DNV-GL also considers the persisting age of the specimen. "Testing of aged cables with voltages higher than nominal can increase the risk of insulation breakdown" (Recommended Practice DNV-RP-J301, p. 128).

2.4 Definitions

Due to the ambiguous wording of the standards, some parameters must first be clarified. Water is examined and a theoretical examination of polyurethane as a test object will take place. For this purpose, a direct limitation is to be made: polymeric materials undergo modifications by additives. A brief overview and a proposal for adapting the test method in this respect can be found in a previous study (Erfurth and Rudorf 2016, p. 22). In the context of this work, a general approach is to be pursued and polyurethane is to be regarded as a pure substance.

For a better understanding and interpretation of the measured insulation resistances, the chemical processes leading to the degradation were briefly presented in section *2.1* and *2.2*. With respect to polyurethane, water trees will be introduced, which are frequently observed as a result of laboratory ageing in particular.

2.4.1 Water

Distilled (and deionized) water can act as an insulator[5], but some water molecules are dissociated, so that few ions are available to conduct the electrical current and make water a bad insulator. Salts in the solid state are - despite their ionic structure - mostly non-conductive. The binding forces between the ions are so high that not enough ions can move freely enough. Dissolved salts, on the other hand, have ions that are no longer firmly bound to their neighbouring ions, and therefor can easily transport the electric current through ion conduction.

The number of H_2O+ and OH^- ions contained in water can be represented by the following reaction equation:

$$2H_2O \rightleftharpoons H_3O^+ + OH^- \qquad (2\text{-}12)$$

The concentration of H^+ and OH^- ions (c_{H+} and c_{OH}) expresses the basicity or alkalinity. The ion product of both quantities gives the dissociation constant:

$$c_{H^+} \cdot c_{OH^-} = K_W \qquad (2\text{-}13)$$

with

$K_W = 1{,}008 \cdot 10^{-14} \, mol^2/l^2.$ Dissociation constant for water at 25°C

The dissociation constant varies with the temperature, because the dissociation of the water is endothermic. As a result, the basicity increases and the acid effect decreases as the temperature rises. At 25°C the concentration of H^+ and OH^- ions is balanced in pure water. According to Hütter (Hütter 1994, p. 45), this leads to a neutral pH value of 7.0. It is further explained that the water reacts differently due to the very low electrolytic dissociation. Depending on the reactant, the reaction product becomes a weak acid or base. This property is called amphoteric behaviour.
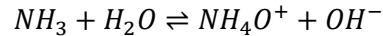
<u>alkali</u>   <u>acid</u>

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[5] Deionized water is produced by removing the charged components from the water. Deionized water has a pH value of 5.0 from Hütter 1994, pp. 81–84.

Water as proton acceptor

$$HCI + H_2O \rightleftharpoons H_3O^+ + CI^-$$
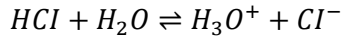
Water as proton donor

$$NH_3 + H_2O \rightleftharpoons NH_4O^+ + OH^-$$

In literature (Grigull 1983; Hütter 1994) the dielectric constant (relative static permittivity) and ion product of water are descriptive parameters of electrochemical behaviour. The purity can be determined with different measuring methods on the basis of electrical conductivity or resistance. As already mentioned, deionized water contains hardly any ions and thus has a low electrical conductivity and a high resistance.

Table 3 Resistivity of water (Lenntech)

	ultra-pure water	pure water	purified water
resistivity	10-18 MΩ.cm	1-10 MΩ.cm	1-0,02 MΩ.cm
conductibility	0,1-0,0555 µS/cm	1,0-0,1 µS/cm	1-50 µS/cm
manufacturing process	Blending bed ionen exchanger	k-times basic blending bettexchange system	basic blending bettexchange system

A study by Ellison (Ellison et al. 1998) updated the measured values of the conductivity of seawater[6]. This investigation showed, that for a NaCl solution with a solid salt content, the change in conductivity as a function of temperature is not generally linear, as presented in Figure 14. But nevertheless, one can assume linearity over a relatively small salinity range.
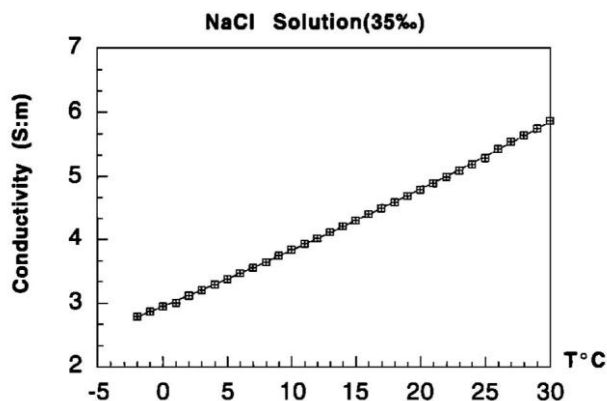

Figure 14: Conductivity of a 35ppt saline solution as a function of temperature (Ellison et al. 1998, p. 643)

The relation between salinity and conductivity is shown in Figure 15 at four different temperatures. For the relatively small salinity range a linearity of conductivity is reasonable. It is assumed a direct proportional increase of the conductivity with the number of free ions dissolved in the water.

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[6] Excursus for differentiation seawater - artificial seawater:
According to investigations by Riley and Skirrow in 1965 (from Ellison et al. 1998, p. 641), the density of the organic content of seawater is in the range of 1.50-3.00 g/m³, which applies to most seawater areas. The particle sizes ranged from $1*10^{-6}$ m to $50*10^{-6}$ m. It was estimated that 14% of particulate suspensions were of organic origin. Organic particles included bacteria, fungi, phytoplankton, zooplankton and detritus.
The inorganic suspensions consisted of different types of mineral particles. Trace elements of various complexes of metals, mercury, cadmium etc. were also detected. The dielectric constant measured in the study showed minimal deviations. However, these were in the range of the measuring error of 1 %.
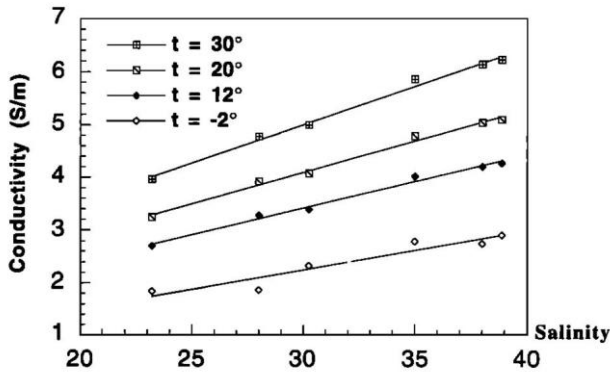
Figure 15: Conductivity as function of salinity at four temperatures (Ellison et al. 1998, p. 643)

Ellison found that there is overall no significant difference between natural seawater and synthetic seawater made using the standard recipe (ASTM D 1141-98). But he proclaimed that there is a significant difference in the conductivity and permittivity between natural seawater and an aqueous NaCl solution of the same salinity. (Ellison et al. 1998, p. 646)

In terms of salinity, Feistel (Feistel 2008b) did a study regarding the validity range of the equation of state of density (with water composition according to ITPS-68) and experimental values of Grunberg (Grunberg 1970). Figure 16 shows curves computed with the Gibbs function, at normal pressure and IPTS-68 temperatures. The numbered symbols represent the Grunberg data for artificial seawater, indicated with 2: 20 °C; 4:40 °C; 6: 60 °C; 8: 80 °C
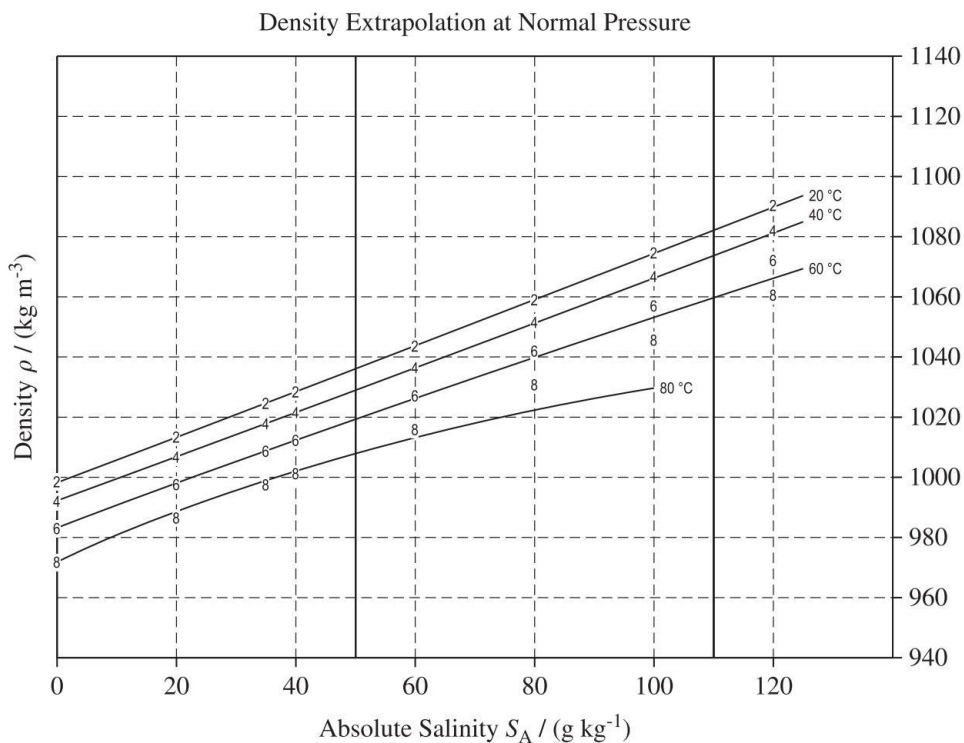

Figure 16: Comparison of seawater densities at high salinities and temperatures (Feistel 2008b, p. 1663)

The graph shows that the empirical values are almost proportional across the entire range. The equation of state is valid in the range of salinity up to 40 g/kg over the entire temperature range, but

with increasing deflection with salinity at temperatures above 40 °C. According to (Feistel 2008b; Ellison et al. 1998) one can assume that the transferability of conductivity for artificial seawater and real seawater are given for a range of salinity up to 40g/kg.

According to Wilhelm (Wilhelm 2008, p. 79), the compound carbon dioxide - water - calcium is of central importance in water chemistry. This compound influences the following factors:

- water hardness (carbonate hardness)
- corrosiveness

Water absorbs carbon dioxide from the surrounding atmosphere until a state of equilibrium occurs. Thus, the carbon dioxide concentration of the environment determines the amount of carbon dioxide dissolved in the water. Finally, the dissolved carbon dioxide reacts with the water and forms carbonic acid.

$2 H_2O + CO_2 \rightarrow H_2O + H_2CO_3 \rightarrow (H_3O^+) + (H_3O^-)$
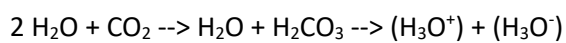
Only for produced distilled water a pH of about 7 can be assumed. As soon as it encounters ambient air a slightly acid pH value is reached within hours.

The dissolved calcium carbonate functions as a buffer. "When water is titrated with calcium hydroxide in a pH range of 4.3 < pH < 8.2, the H^+ ions from the first carbonic acid dissociation stage are bound by the OH^- ions of $Ca(OH)_2$ to undissociated H_2O; this causes the carbonic acid to continue dissociating until it is completely dissociated at pH 8.2. The resulting H^+ ions will be completely bound to undissociated water, leaving the $HCO_3{}^1$ anions" (Wilhelm 2008, p. 82). However, it must be noted that at pH values > 8.2 the fraction of $CO_3{}^{2-}$ ions increases continuously.
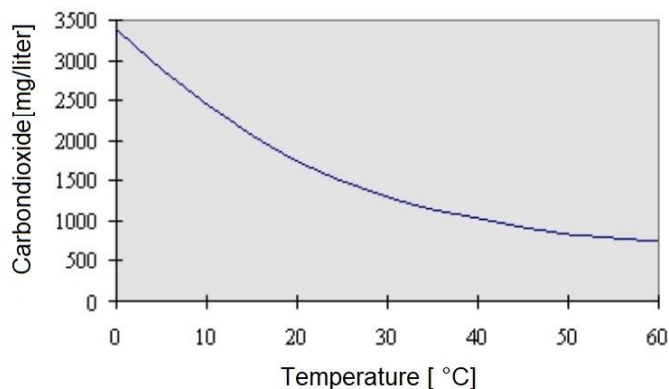

Figure 17 Solubility of carbon dioxide in water depending on temperature

In terms of temperature dependence, the reactions shift at elevated temperatures, as can be seen in Figure 17 from the proportion of dissolved carbon dioxide in water. Further the solubility of gases does depend on temperature and pressure, this results in the properties of seawater being are affected.

The overall properties of seawater depend on salinity, pressure and temperature. Based on measurements Safrov (Safarov et al. 2012) extends the existing model to adjust for higher temperatures and for pressure up to 140 MPa. Starting from the extension of the current equation of state (Feistel 2008a) a model was developed which is valid for:

- Absolute salinity S_A=31.68370 - 55.52861 g/kg
- Temperature   T=273.15–468.15 K
- Pressure   p= 0.101 – 140 MPa.

The interconnected relations are illustrated using the example of density in Figure 18. The displacement due to temperature increase is also superimposed by a displacement due to the change in pressure.
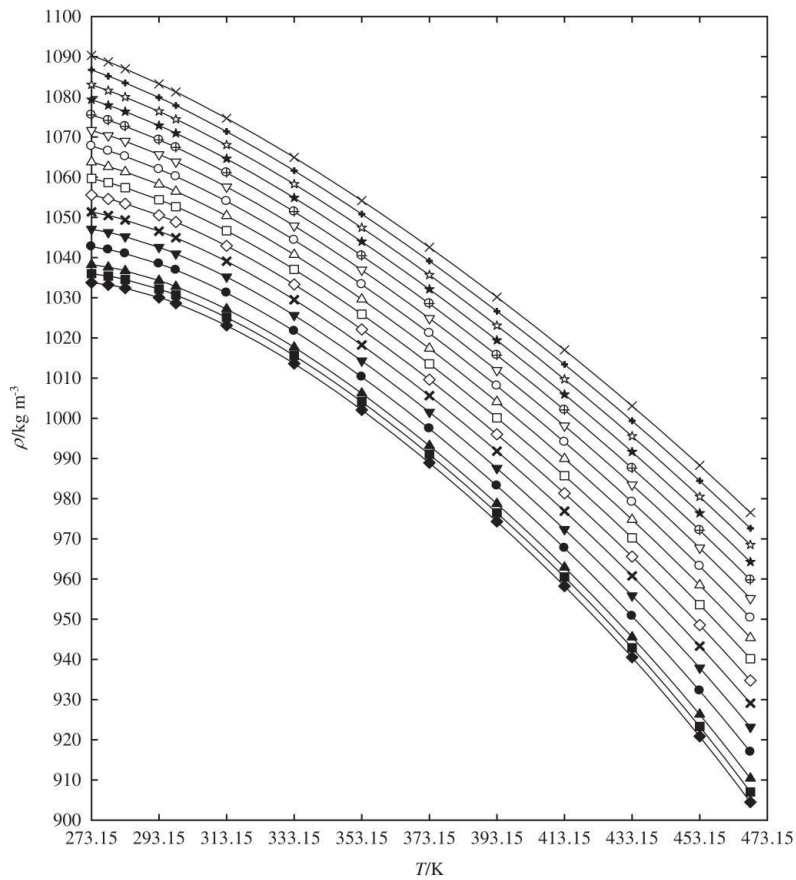
$$\rho = f(S_A, T, p) \tag{2-14}$$

Fig. 2. Plot of density ρ of seawater $(S_A=41.825\ \text{g kg}^{-1})$ *vs* temperature T calculated by Eqs. (1)–(4): ♦, 0.101 MPa (after T=373.15 K—p_s of water); ■, 5 MPa; ▲, 10 MPa; ●, 20 MPa; ▼, 30 MPa; ×, 40 MPa; ◇, 50 MPa; □, 60 MPa; △, 70 MPa; ○, 80 MPa; ▽, 90 MPa; ⊕, 100 MPa; ★, 110 MPa; ☆, 120 MPa; +, 130 MPa; x, 140 MPa.

Figure 18 Density of seawater vs temperature and pressure, with indication (Safarov et al. 2012, p. 150)

In Figure 19 both, the equation of state (EOS) for determining density and the practical salinity scale (PSS 78) are plotted together (in light grey). One can see that these equations are accurate only to a certain point. A further approach was taken by Feistel (Feistel 2008a), who describes the properties of seawater by the specific Gibbs energy[7] of seawater. He compares the results of the theoretical

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[7] The Gibbs function permits the consistent computation of all thermodynamic properties of seawater from a single expression. The Gibbs energy of a seawater sample containing the mass of water, m_W, and the mass of salt, m_S, at the absolute temperature T and the absolute pressure P. Feistel 2008b, pp. 1641–1642.

approach with the empirical data for artificial seawater. Another comparison was done by Safarov et al. (Safarov et al. 2012), who used the empirical data of seawater and did an adjustment for the model to describe higher salinities, temperatures and pressures. A proportional relationship between density and conductivity can be seen, as it is given to a sufficient extent.
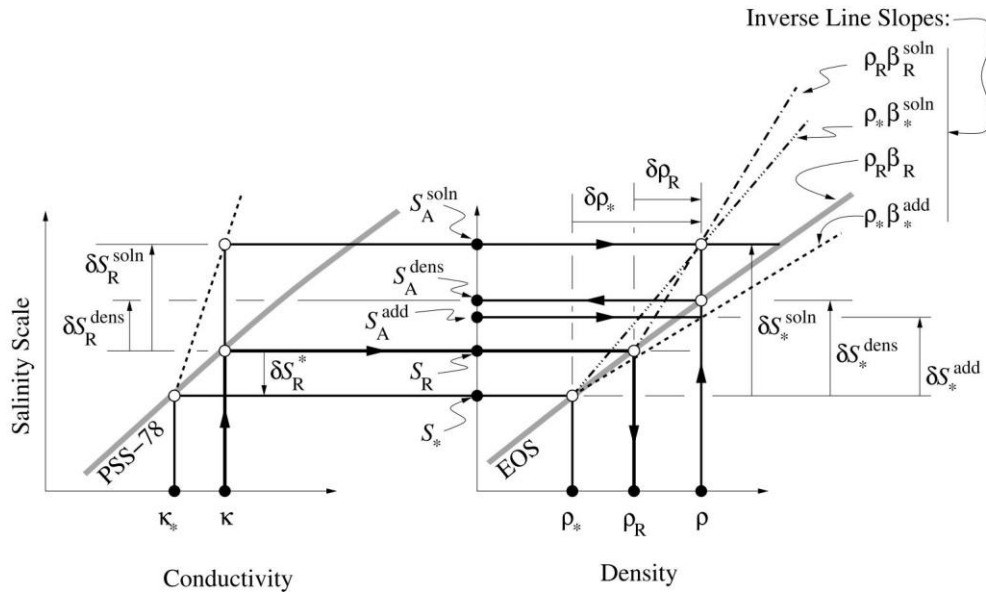

Figure 19: Schematic illustrating the relationships between different salinities, densities, and conductivities for standard seawater (SSW) (Pawlowicz et al. 2011, p. 369)

Other biochemical peculiarities occurring, under the change of pressure, were noted by Pawlowicz (Pawlowicz et al. 2011, p. 384). He found out, that a growth in dissolved inorganic hydrocarbons occurs in seawater, with increasing pressure. These dissolved carbons come from segregation of CO_2 during the remineralisation of organic carbon. And as well, when $CaCO_3$ dissolves under increased pressure, it dissociates and is deposited as CO_3^{2-}. The greatest change in the composition of seawater has been observed in the increase of HCO_3. The transformation of organic nitrogen to inorganic also produces HNO_3, which splits into H^+ and NO_3^-. The dissolution and hydration of solid SiO_2 expands with increasing temperature, which leads to an elevation of the concentration of $Si(OH)_4$.

"However, unlike the case for many of the laboratory experiments, corrections that arise from conductivity changes result in a much smaller discrepancy because the products of ocean biogeochemical processes are relatively unconductive. Potentially this makes conductivity/salinity/density relationships to be used in reducing ocean measurements much more complex" (Pawlowicz et al. 2011, p. 385).
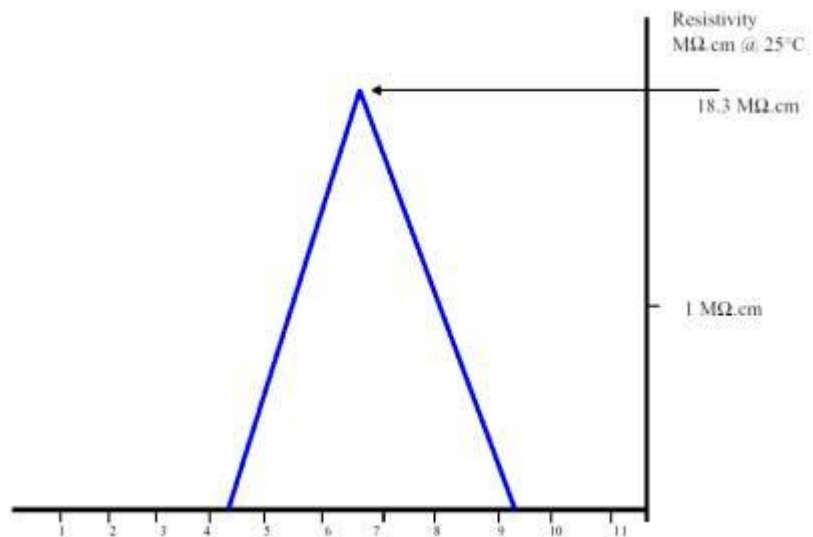

Figure 20 pH value for ultrapure water by determining the electrical resistance (Lenntech)

To conclude, the relation between resistivity and pH level (as in Figure 20) cannot be expressed as a linear function as in pure water.

Water with regard to electrolysis

Seawater has dissociated ions which are an electrically conducting solution when a current is applied and can be called an electrolyte. "The electrically neutral condition states that the sum of all cation equivalents or cation equivalent concentrations in all waters must be the same as the sum of all anion equivalents or anion equivalent concentrations. This means that all waters are electrically neutral " (Wilhelm 2008, p. 103). In a nutshell, when water is used as a conductor, the balance changes and chemical reactions are induced. These chemical reactions can involve the specimen, the electrodes or the water itself. Another chemical reaction is hydrolysis. Hydrolysis is known to be the most important reaction associated with the loss of the mechanical properties of polymers. (Laycock et al. 2017, p. 151).

2.4.2 Polyurethane

The samples examined in this study are cables with the sheath material polyurethane. Polyurethane is the product of polymerization reaction. Reaction polymers can be described as propagating reaction, where a catalyst is attached to a positively charged hydrogen atom of a hydroxyl group of the diol. Caused by the strong hydrogen bond (shown in Figure 21 as a blue dotted line) the polarization is shifted from the hydrogen atom to the catalyst and further away from the oxygen atom. Due to the greater distance of polarized molecules, the oxygen atom has an excess of electrons and bonds to a carbon atom of the isocyanate group of the diisocyanate. The connection of the two monomers can be seen in the left part of Figure 21, the two are shown perpendicular to each other.
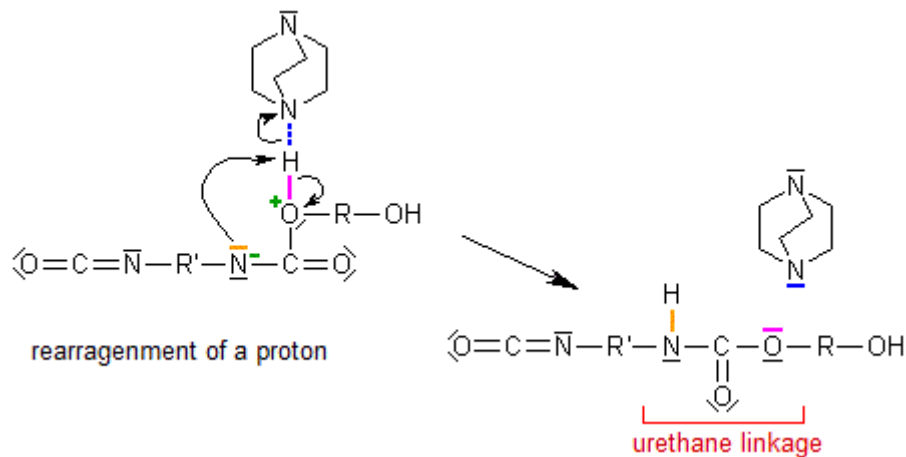
Figure 21 Urethane linkage (Ibold and Brockmann 2015)

By combining the oxygen atom with the carbon atom, one of the electron pairs of the carbon is instantly shifted to the combined nitrogen atom. This results in a negative partial load of nitrogen. The free pair of electrons pulls the hydrogen atom to the nitrogen atom, whereby the catalyst is detached and the hydrogen is bonded to the nitrogen atom as a proton. The remaining electron of oxygen pairs with the compound carbon into a double-bridge bond. This grouping is called urethane linkage. At one end there is an isocyanate group and at the other end a hydroxyl group. The bifunctional molecule allows the process to be repeated and the chain to continue growing. The degree of cross-linking of the polyurethane varies depending on the number of groups present. High cross-linking makes the compounds hardly soluble and influences their mechanical properties.

Polyurethanes consist of crystalline and amorphous areas. They have a melting temperature for the crystalline areas and a glass transition temperature for the amorphous areas.
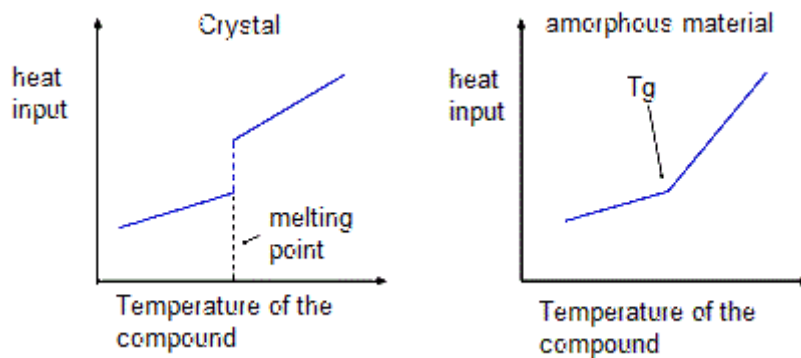

Figure 22 Glass transition and melting point (Kratzert)

The glass transition temperature depends essentially on the structure of the polymer. In general, polyurethane is a non-polar dielectric(Babiel 1985). When T_G is exceeded, not only the mechanical properties, but also the electrical properties change, the latter to a non-linear extent, due to the changed gradient of the molecular chains' elbow-room.

According to Ahmad (Ahmad 2012, pp. 6–8) the temperature directly affects dielectric properties. With increasing temperature thermal agitation is enhanced and the intermolecular forces between polymer chains are broken. The polar group will orient increasingly freely, allowing the molecules to keep up

with the changing electric field. When the temperature exceeds the glass transition temperature, the dielectric constant is again reduced, due to strong thermal motion which disturb the orientation of the dipoles. For lower temperatures, the segmental motion of the chain is congealed and thus, the dielectric constant will be reduced too.

Depending of the temperature the relaxation process will be different. "The dielectric loss will show maxima at respective relaxation mechanisms as the temperature is increased […]:

- α relaxation: Micro-Brownian motion of the whole chain. Formally this motion is designated as glass transition.
- β relaxation: Rotation of polar groups about C-C bond e.g. CH_2Cl and $-COOC_2H_2$ conformational flip of cyclic unit.
- γ relaxation: libration of phenyl ring and limited C-H segmental chain movement." (Ahmad 2012, p. 7)
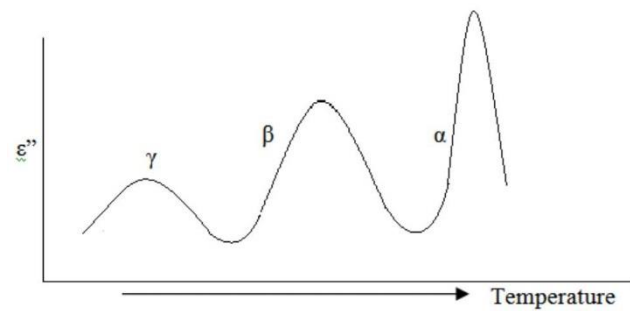

Figure 23 schematic dielectric loss curve for polymer as temperature is increased (Ahmad 2012, p. 8)

The relationship between the degree of crosslinking and the properties of polymer materials was investigated and modelled by Rahmati 2012 (Rahmati et al. 2012). The density of polyurethane membranes varies depending on the degree of polymerization. The density of PU with a degree of crosslinking 5 obtained by simulation is 1.027 g/cm^3, which is highly consistent with the experimental value of 1.04 g/cm^3.
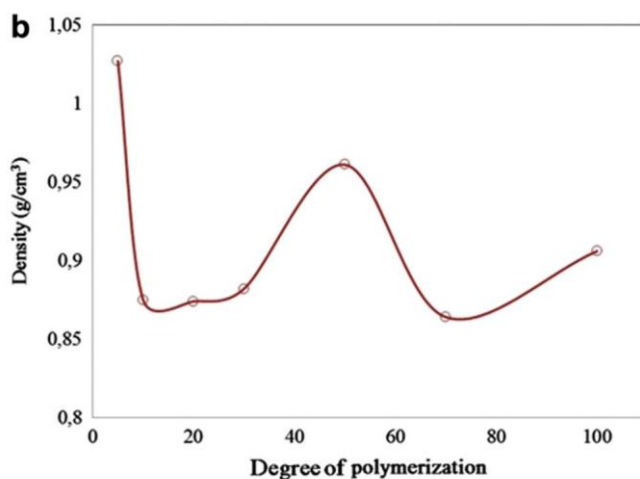
Figure 24: density of polyurethane (Rahmati et al. 2012, p. 1944)

As can be seen in Figure 24, the density of the polyurethane membrane is reduced by increasing the degree of polymerization. However, it increases again at degrees of polymerization between 20 and 50. This is explained by Rahmati (Rahmati et al. 2012, p. 1944) by the folding of the chain forms. A similar explanation was found for the

Figure 25 folding, i.e. influencing the helix–coil (Yu and Hecht 2016)

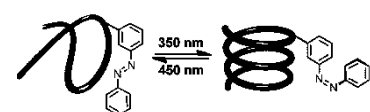decrease of the fraction free volume[8] with an increasing degree of polymerization. Except the decreasing volume fraction at the degree of polymerization of 50, it is associated with the folding of the polymer chains. For PU 70, the volume fraction increases again because the chains are long enough to fold and rotate again.
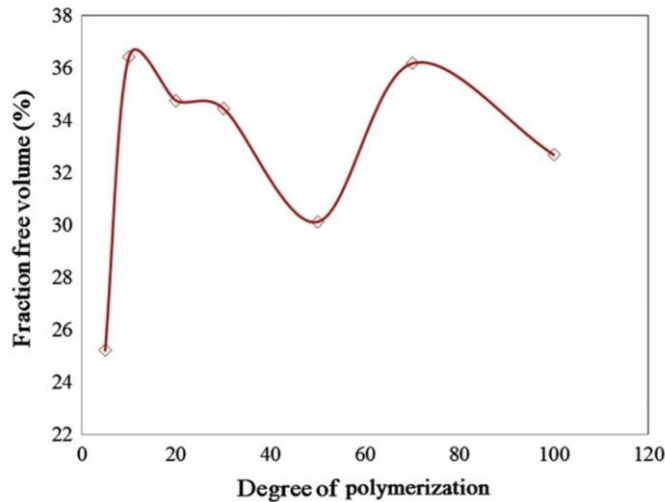

Figure 26: The change of fractional free volume of polyurethane membranes with the degrees of polymerization. The solid lines are just for seeing the better trend. (Rahmati et al. 2012, p. 1945)

A more detailed description of the connections between the degree of polymerization and thee-dimensional formation cannot be given here, since an adequate presentation of this theory is not possible in the limited context of this work. However, it becomes clear how complex the structure of polyurethane can be and that increasing the degree of crosslinking has an influence on the properties of polyurethane.

2.4.3 Dielectricity

According to Ose (Ose 2013), polarization is induced when there is electric field applied onto the materials. In the absence of electric field, the electrons are distributed evenly around the nuclei. As the electric field is applied the electron cloud will be displaced from the nuclei in the opposite direction of the applied field. A separation of positive and negative charges will be the result and the molecules behave like an electric dipole. There are three different types of polarization:

- Electronic polarization: minor displacement of electrons with respect to the centre of charge
- Atomic polarization: asymmetric distortion of atomic position in the molecule or lattice
- orientation polarization: in polar molecules (water molecule) have a tendency for permanent dipole to align by the electric field, to give a net polarization in that direction

Only when a static electric field is applied on to an insulator the dipoles become permanently polarized giving a dielectric constant as ε_r. If an alternating electric current is applied and the field changes, polarization will oscillate with the changing electric field.

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[8] Fractional Free Volume (FFV) is an empirical dimensionless parameter that characterizes the free volume in polymers. FFV can be defined as the ratio V_f/V_{sp} , where V_f (cm3/g) is the free volume and V_{sp} (cm3/g) is the specific volume of the polymer, the reciprocal density ρ. Yampolskii 2015, p. 1.

Thus, the electronic polarization is instantaneous, as it is able to follow in phase with the changing electric field. In comparison, atomic polarization is able to follow the oscillating electric field better than orientational polarization.

"When an electric field acts on a dielectric with $\varepsilon_r > 1$, the centre of charge of a non-polar dielectric in the atomic lattice is shifted (polarization) and dipoles are formed" (Ose 2013, p. 136). To measure this effect a current can be applied. The method is not fundamentally different from that of a measurement of a conductor. One of the differences is the relevance of the duration of the applied voltage as an important parameter. In some cases the voltage gradients are also important. When the voltage is applied to the sample, the current usually drops asymptotically to a threshold below 0.01. This decrease in current over time is due to dielectric absorption. The ratio of current and time can be drawn for comparative purpose:

$$I(t) = A \cdot t^{-m} \tag{2-15}$$

with

A	Constant, defines the current at time unit
m	Material factor $(0 - 1)$

Ahmad describes (Ahmad 2012, pp. 3–4) that some elements and structures display a higher polarizability than the others. Iodine, sulphur, bromine and aromatic rings are considered as highly polarizable. This leads to an increase in dielectric constant if molecules of these groups are present.

For large atoms like iodine and bromine the electron cloud is so big and further apart from the attraction of the nucleus. Hence, these atoms display a high polarizability due to their size. The opposite with small atoms like fluorine, which has a relatively small atomic radius and a negative charge. With the ability to hold the electron cloud so much tighter it results in a low polarizability. And as a consequence, it will cause a lower dielectric constant.

Another determining factor for the dielectric constant is free volume. The presence of pores as a form of free volume results in a decrease in dielectric constant. These spaces can be occupied by air whose relative permittivity[9] is about one. The fractional free volume is related to the density of the material, as shown before in *Polyurethane*. A high free volume will result in a lower density and thereby a smaller polarizable group per unit volume. A variance introduced by Ahmad (Ahmad 2012, p. 5) is the replacement of hydrogen with fluorine, which results in lowering of the dielectric constant because fluorine occupies a higher volume.

2.4.4 Diffusion

In general, the process of diffusion depends on the temperature and leads to mass transport. (Bargel et al. 2008, p. 27). The transport process of gaseous and liquid substances itself depends according to Koldrack (Koldrack 2009, p. 22) on several factors:

- Polymer structure (interactions between the individual polymer chains)

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[9] The permittivity for liquid water at atmospheric pressure and T = 273.15 °K is ε = 87.903455 uncertainty 0.04 Feistel 2008b.

According to Laycock it is important to consider factors that control uptake in complex matrix environments, such as the polymer's free volume, the physical state of the polymer, and the glass transition temperature. (Laycock et al. 2017, p. 155)

- Temperature (activation energy)

  For a transferable statement regarding the diffusion rates with temperature changes, knowledge of the activation energy is mandatory. Polymers do not generally have constant activation energy, e.g. polychloropenes, has no constant activation energy between 25 °C and 95 °C. The activation energy should therefore be determined for the relevant temperature interval. (Koldrack 2009, p. 33)

- Molar size of the diffusing substances

  The atomic diameter of water can be determined from interpolation of the effective ionic radii of the isoelectronic ions of O^{2-} (2.80 Å), OH^- (2.74 Å) and H_3O^+ (2.76 Å). Coincidentally, this diameter is similar to the length of a hydrogen bond. The water molecule (bond length 0.96 Å) is very small, with only H_2 (bond length 0.74 Å) being a smaller molecule. The water will penetrate through in the amorphous regions, resulting in more rapid hydrolysis in the amorphous regions. But the effect of molecular weight on water uptake is mostly theoretical and based on limited studies to date. (Laycock et al. 2017, p. 155).

- Interaction between polymer and medium

  "The increase in pore volume following degradation, as well as the increase in polymer mobility and the decrease in glass transition temperature (T_g), all allow increased water uptake. […] diffusivity is highly influenced by swelling, not degradation" (Laycock et al. 2017, p. 155).

In general, diffusion can be viewed through two approaches

1. mechanisms of fluid penetration and penetration with focus on transport kinetics and diffusion mechanism

2. interaction between polymer and medium (chemical degradation and removal of polymer components)

Past investigations by Rudorf, Koldrack and Erfurth followed the first approach. The problem is that by hydrolysis the composition of the adsorbent surface varies and thus the assumptions of a constant diffusion constant relevant for the arrhenius equation are not given in the solvate range. The two mechanisms of diffusion are coupled and are usually expressed by a constant diffusion constant.

2.4.5 Adsorption

Adsorption is an interfacial phenomenon and it occurs in the context of solids with a large internal surface. As the distance from the adsorbent surface increases, the pressure and density change

relatively. In general, a distinction is made based on the strength of the interaction between physisorption and chemisorption.
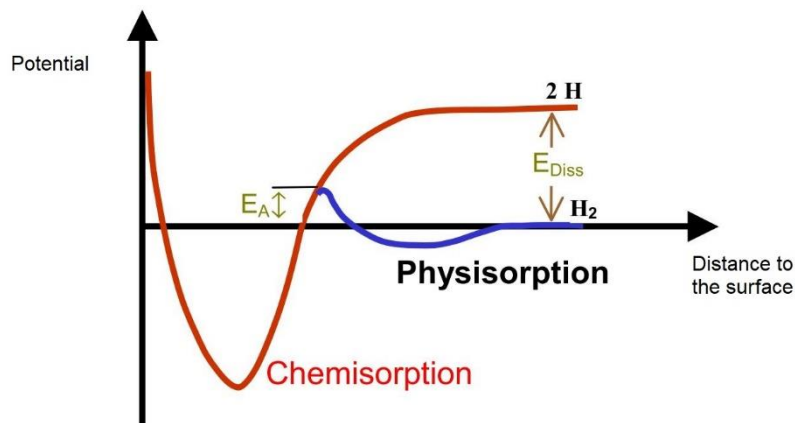

Figure 27 Potential curve of a hydrogen molecule approaching the adsorbent surfaces (Brehm 2009, p. 4)

Depending on the pore structure of the adsorbent, a monomolecular layer forms during chemisorption. According to Langmuir's theoretical model, all places on the surface are occupied by one molecule each, practically there is also a multi-layer adsorption within the physisorption.

Table 4 Comparison Physiosorption/Chemiesorption taken from (Brehm 2009, p. 4)

Parameter	Physisorption	Chemisorption
Adsorbent	all solids	only specific solids
Adsorbate	all gases below their critical temperature	some chemically reactive gases
Temperature-ranges	low temperatures	usually high temperatures
Adsorption heat	small ($\approx \Delta h_{Condensation}$)	high, corresponds to heat of reaction or dissociation energies
Coverage	Multiple layers possible	monolayer
Reversibility	complete, reversible	often irreversible (Cat. Desactive.)
Surface analysis	Determination of the surface (BET) + pore distribution	Determination of the area of the active centers + reaction kinetics

With regard to the energy potential, weak forces are effective during physisorption and up to 50 kJ/mol are released. Since chemisorption begins after overcoming the activation energy, energy > 100 kJ/mol is released. The boundary layer is polarized by adsorption. "The electric field enhancement on the surface of sheath in both hydrophobic and hydrophilic cases observed through simulation suggests higher discharge activity and could result in electrohydrodynamic force that helps moisture ingression into the body of insulator" (Yashodhara et al. 2017, p. 5).

An extensive study (Rahmati et al. 2012) has shown that water adsorption in polyurethane membranes is almost temperature-independent (see Figure 28). This insight can be adopted. The increase in temperature has no significant influence on the percentage sorption of water in polyurethane membranes. But the increase in temperature increases the thermal mobility of the polymer according to the theory of free volume, which in turn results in additional cavities that can increase the flow. In

summary, polyurethane does not absorb more water at higher temperatures, but faster. As a limitation, it must be noted that this is only applicable for pure substances:

„adsorption in polyurethane membranes is a function of membrane free volume, its degree of polymerization and the size of sorbate molecules." (Rahmati et al. 2012, p. 1947)
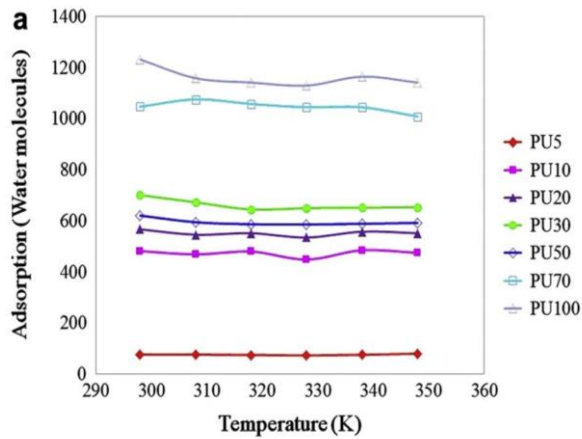

Figure 28: The adsorption isotherms of water and furfural as the function of temperature in PU membranes at 298-350 K and 10 bar for feed composition 8.42% mol for water (Rahmati et al. 2012, p. 1948)
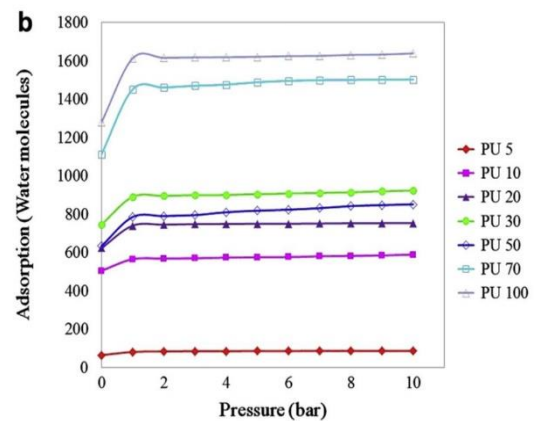
Figure 29: The adsorption isotherm of water in PU membranes at 298 K and 0-10 bar. (Rahmati et al. 2012, p. 1946)

The water adsorption is increasing up to a pressure of 2 bar and then changes only moderately up to 10 bar.

2.4.6   Hydrolysis

The following Figure 30 shows the different types of diffusion and hydrolysis schematically according to Laycock. Figure a) shows a surface erosion with growing hydrolysis front (e.g. enzymes), Figure b) a mass erosion with autocatalysis by retained degradation products (e.g. PLA) and Figure c) a mass erosion without autocatalysis. L_{crit} is the critical sample thickness, λ' is the first order pseudo-hydrolyse rate and D the diffusion coefficient. It should be noted that "erosion" refers to the loss of polymer material when oligomers and monomers leave the polymer. The illustration shows the processes only schematically. The complete erosion of a polymer takes considerably longer than the loss of tensile strength due to the splitting of the polymer chain.
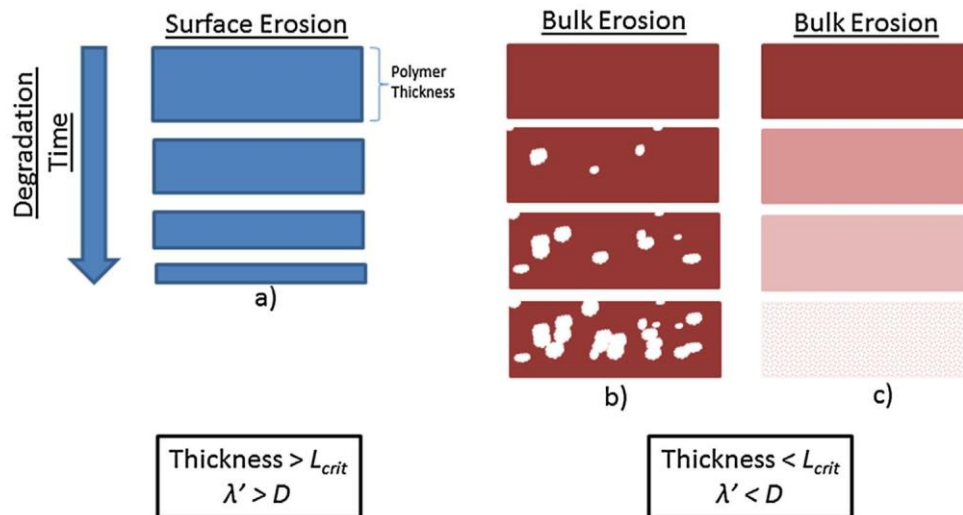
Critical sample thickness $L_{crit} = (D/\lambda')^{1/2}$, D is the diffusion coefficient of water and λ' the pseudo first order hydrolysis rate constant

Figure 30: Schematic illustration of three types of erosion phenomena: (Laycock et al. 2017, p. 156)

Common to the oxidative and hydrolytic degradation processes is the decreasing degradation rate with increasing degree of crystallinity. In hydrolytic degradation, this is due to the fact that water cannot easily penetrate the high-order crystalline areas of the polymers.

For maintaining the mechanical properties key is whether degradation takes place as a surface process or as a mass process. As known from previous studies (Erfurth and Rudorf 2016), the composition of the polymer material is the regulating part in the relative diffusion and hydrolysis rate. This is divided into the consumption phase (a), during which the stabilizers are degraded, the short transition phase of the induction time (b), during which property changes begin and the subsequent degradation (c). The last phase ends when the limit values are reached and the service life ends.
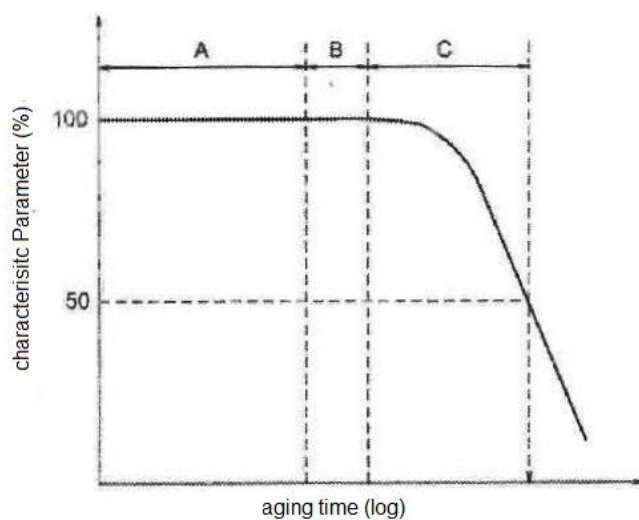

Figure 31 The three phases of polymer lifetime (Koldrack 2009, p. 19)

The type of bond, present in the main polymer chain, defines the kinetics of bond cleavage. Although, the kinetics of these reactions can change significantly due to the presence of catalysts, or through the influence of adjacent groups, such as through areal and electronic effects.

The interdependency between the rate of water diffusion in a polymer and the kinetics of bond cleavage caused by hydrolysis, plays a key role in the mechanism of degradation of polymers (Laycock et al. 2017, p. 155).

2.4.7   Water tree

Hydrolysis can cause although water trees, which reduce the dielectric strength of cables. They are one of the main causes of failures of older cross-linked polyethylene constructions with (XLPE) sheathing of medium voltage cables that have only been in use for 5 to 15 years (Densley 2012, p. 1). An above average number of bow-tie water trees (see Figure 32, down) occurs on laboratory aged cables. One assumes the cause is the high ageing voltage in combination with an increased moisture content due to pre-treatment in a water bath at 55 °C (IEEE, p. 43).

The methods of insulation diagnostics are based upon the assumption, that water tree formation of the insulation influences the overall dielectric properties, i.e. conductivity and polarisation. If water trees are present in sufficient numbers they have a n enduring effect on the dielectric properties of the cable system. Water trees are a locally degraded phenomena. They can completely bridge the insulation thickness and occur often in low voltage cables, and in cables with a low insulation thickness of less than 5 mm in particular. Bridging water trees are electrically much easier to recognise than non-bridging water trees, because the direct current flowing through the insulation on the water tree can be up to three orders of magnitude larger ($1 \geq$ µA) than the current of a non-bridging water tree (~1 nA).

In the majority of cases, water trees occur in polymeric insulations that are exposed to an alternating electric field and a relative humidity of over 75%. The name water tree comes from the fact of the tree-like structures, see Figure 32, which consist of rows of cavities, so called "pearl chains". The cavities of these can vary from about 0.1 µm to 5 µm in diameter. In most cases

Figure 32 Typical water trees growing from conductor screen (above) and from a contaminant within the XLPE insulation (Technical Brochure 493, p. 6)

two techniques using direct current are applied to detect both bridging and non-bridging water trees, one is the polarisation/depolarisation current method and the other a leakage current method (Technical Brochure 493, pp. 5–9).

The leakage current through a bridge water tree is not uniform for negative and positive polarities of the DC voltage, as shown in Figure 33. Under positive and negative polarity the tips of the water trees have different charge injection and extraction characteristics, because they act as needle electrodes. Based on the characteristic of this phenomenon and the assumption that this behaviour occurs in AC waveform at different half-cycles, too, one applies an on-line method on lower voltage cables. A combination of a low pass filter and a DC ammeter are connected into the ground lead and one
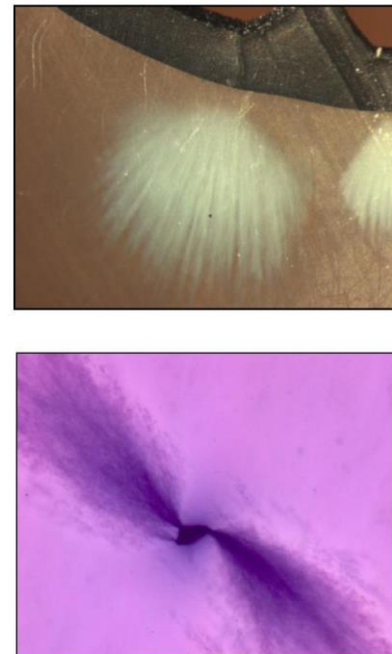
measures the DC component of the AC waveform. "Laboratory tests on service-aged cables have shown that the AC breakdown voltage decreases with increasing DC component current. Any current due to local corrosion cells may have an influence on the component." (Technical Brochure 493, p. 26)

In IEEE 43 (IEEE 43, p. 3) is a phenomenon with similar characteristics described, which is related to the presence of moisture: Electroendosmosis. This is an effect that is occasionally observed in older insulators. Different insulation resistance values are achieved if the polarity of the test leads is reversed. Typically, the insulation resistance with positive polarity (the conductor is connected to the positive pole) is considerably higher than with opposite polarity.
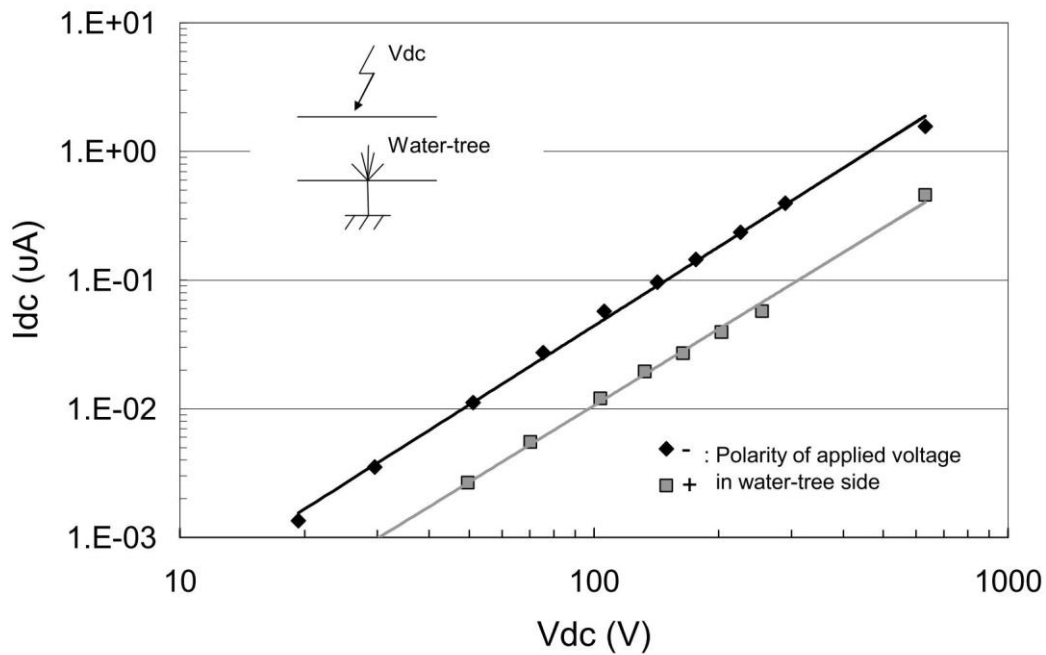

Figure 33: effect of voltage polarity on DC leakage current (Technical Brochure 493, p. 26)

2.5   Arrhenius adjustment with E_a

The approach in standard DIN EN 60216 can be described as the conventional method for determining the long-term thermal behaviour. The uncertainty of the long-term behaviour estimated using the Arrhenius approach is expressed in very high safety margins. VG 95218 speaks of the existence of the service life verification if the "temperature curve, which is 20 °K above the service life and does not reach the failure criterion after one year". (VG 95218-2, p. 102). According to Rudorf's projection, using the extension factor (Rudorf 2016a, p. 37), this means that the failure criterion can be exceeded at the earliest after 4 years for the service life verification.

In order to increase the accuracy of the prediction of the service life, VDE 0304-7-1 proposes a further evolved method. Modern thermodynamic test methods such as dynamic differential calorimetry (DSC) and thermogravimetry (TGA) are used to determine the activation energy E_a of the material. Among other arguments, this has the advantage that the glass transition can be easily distinguished from other effects (NETZSCH-Gerätebau GmbH 2013, p. 28). In addition, the temperature interval and the maximum storage temperature can be determined very precisely. Based on the association of the "activation energy correlates with the HIC of thermal resistance" (DIN EN (Vornorm) 60216-7-1, p. 5), thus, a 1-point determination of the long-term thermal behaviour according to DIN EN 60216-5 can be carried out. The ageing regression is determined directly over the highest temperature. The

temperature index is determined due to the continuing uncertainties regarding the precision of the Arrhenius approach via the detour of the relative thermal resistance index (RTE[10]). At the present state of knowledge, this method has not been validated, which is the reason why VDE 0304-7-1 is marked as draft.

2.6 Presentation of the content of the paper

Die unter 2.4.1 dargestellten Erkenntnisse insbesondere von Feistel

The findings presented in *2.4.1 Water*, in particular Feistel (Feistel 2008b) show that for laboratory tests the conductance of water on the temperature and time axes is variable. To what extent there are interactions between sample and medium is not the subject of this study. It should be taken into account that the measured insulation resistance including variable resistance of the water. Koldrack cannot substantiate no indication about the influence of the frequency of water changes on the result (Koldrack 2009, p. 59).

With regard to the investigation and measurement, there is a differentiated factual situation in homolytic cleavage. The formation of radicals takes place preferably in a non-polar environment, since the formation of ions in the polar environment is often favoured. The applied current creates a polar environment and thus the measurement influences degradation.

Cigré regularly produces a research report on the causes of damage to cables. The conclusion is that hydrolysis does not play a role in cable failure. And the influence of water also plays no role, since the degradation does not appear to be chemically caused (Technical Brochure 279).

Rudorf proposed a calculation method to compare the measured values from the short-term bearings with those of the base curve. The measured values of the time to failure to be regarded as provisional are to be corrected by comparing the calculated values with the values from the long-term storage - the basic characteristic.

Adjustment function by Rudorf: $t_N = t_R + R^k \cdot t_E$ (2-16)

with

t_N	Service life at normal temperature
t_R	Period of time under normal temperature conditions
t_E	Period of time under elevated temperature conditions
R	Extension factor (previously 2)
k	Temperature coefficient with $k = \frac{T_{elevated\ temperature} - T_{at\ normal\ terms\ of\ usen}}{10}$

This makes it possible to evaluate the measured values and to predict the estimated service life more precisely. It can be called an improved correction, due to the utilization of significant milestones (90%,80%,70%,...) of the short-term storage and the base curve, which are shorter in comparison to application of a final milestone in formula (2-13) (Rudorf 2016b, p. 55). The classical method calculates R_Correction only once, at the end of the test. The improved approach is calculating the correction factor

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[10] Definition of the RTE: "numerical value of the temperature in degrees Celsius at which the estimated time to failure of the test material is the same as the estimated time to failure for the reference material at a temperature corresponding to its set thermal life (ATE)" DIN EN 60216-5, p. 5.

continuously and the corrected extension factor is fine-tuned with each further measured resistance as the test duration progresses, thus making the prediction more precise. In contrast to the classical method, in which the evaluation is only possible once the real service life has been reached, the specification of the accelerated method starts already at 90 % of the limit value. For the information obtained with regard to the insulation resistance measurement, please refer to Table 5

2.7 Discussion of different approaches and results

Overall, the standards and recommendations are largely ambiguous, and one alone is not suitable to define framework requirements for the specific application of measurement of insulation resistance at elevated temperatures in water. Deionized water is preferred in some standards, who are dealing with accelerated ageing for simple approximation methods as with the conductivity and pH value, and because of the belief, to have a more legitimate environment for the Arrhenius-equal-behaviour. Which is wrong, hydrolysis is a chemical reaction of an order higher than one, in any case. Furthermore, the density of seawater is changing in dependence on the depth below sea surface. In addition the process velocity of hydrolysis is different for seawater and pure water. Among other factors, Arrieta explains the higher velocity of hydrolysis in deionized water with fewer layers, which are formed around the polyurethane in the process of adsorption (Arrieta et al. 2011, p. 1418).

Water:

The shown scientific studies prove that measurements either with artificial seawater produced according to ASTM 1141 or with deionized water guarantee the best reproducibility. Artificial seawater is recommended as an inlay medium, as the water represents a realistic application during the tests. Also due to the differences shown between NaCl solution and artificial seawater, the seawater is recommended according to the composition of a standard such as ASTM 1141. The assumption that it tends to electrolysis initiated by the measuring current is confirmed. The influence on the measurement and the polyurethane sample is unknown.

Requirements for insulation resistance measurement

VG 95218-2 can be regarded as a representative document for the situation. It refers to a total of 111 other standards and regulations. It does not describe any methods, but rather the VG 95218 is to be regarded as a mere requirements document. An overview about the interconnectedness is presented in BLA. Excerpt from (VG 95218-2, p. 88):

> „Test in water according to DIN EN 50395
> The type standard shall specify:
> Insulation resistance;
> temperature of the water bath."

In contrast to the usual specifications for measurements on one or a few binding standards, that precisely define the measurement method, a large amount of standards must be cited, since the structure is represented inadequately and hardly any boundary conditions are formulated. The temperature adjustment should not be linear, but exponential, due to the change in the material structure and the associated property changes of the polyurethane as well as the effects on artificial ageing. The samples are subjected to multiple loads during storage and measurement and it is assumed that Bow-Tie water trees occur more frequently during artificial aging, than in real applications.

The time interval between measurements varies greatly in the given standards. There is a very conservative demand for a time interval of at least 4 hours. The charging time can be set according to the purpose of the measurement. Current is applied between 1 minutes and 30 minutes for measurement, whereby the length of the charging time with current influences the orientation of the molecules. For the determination of dielectric properties it is recommended 10 minutes and for point measurement 1 to 2 minutes. The range of the voltage to be applied for low voltage cables is specified in the standards in accordance with 80 to 500 V and with 500V recommended.

In general, the arrangement of the samples requires that a certain length of the cable piece not exposed to water. The average length outside the water should be 250 mm. The total submerged length varies and there is no information given on the spatial arrangement. The same applies to the design of the measuring circuit; no clear polarization is recommended. In order to reduce measurement errors, it is pointed out that phenomena such as electrophoresis during the polarity reversal of the measurements cause deviations, due to the surface structure of the sample and the measurement values may therefore be different. With regard to adsorption, the boundary layer is already polarized; it is not known to what extent it is beneficial to apply an electric field for amplification or reversal of polarity and a decay.

To minimize faulty measurements due to leakage surface currents, measurement with applied Guard is generally recommended. The three-point measurements correspond to the current state of the art and can considerably reduce the sources of error, with regard to surface currents. With consideration to the arrangement, the use of a so-called guard ring is recommended. A contact to the guard connection is made at the respective cable ends located outside the water.

The measurement of the residual charge is more complex, but is recommended, too. Since the polarization of the dielectric can be regarded as an indicator of the condition of the insulating material. Considering the conservative intervals of 4 hours between the measurements, the measurement of the residual charge can be neglected in relation to the insulation resistance measurement.

The initial value is very important, as the change in condition is described as a fraction of it, i.e. the ratio of the most recently measured insulation resistance to the initial value. There are also large differences in the set time, at which the initial value is determined as prescribed by the standards. Most of the deviations are caused by different pre-treatments. The determination that the starting value should be determined 48 h ± 6 h after the water deposit is very conservative, but most reasonable in comparison to the others.

3   Pointing out the scientific knowledge deficit

From the presentation of the standards and the description of the chemical processes of water it becomes clear that the Arrhenius context must be adapted for artificial ageing in water. In the context of artificial ageing higher order chemical reactions take place, in contrast to the first order chemical processes of ideal gases for which the Arrhenius equation is proved. Furthermore, several Arrhenius processes take place in parallel, instead of a single Arrhenius process, as specified for the calculations in the standard.

In summary, the following factors are not sufficiently formulated for a reliable further development of insulation resistance measurement:

to be investigated	Material	level of knowledge
yes	Temperature (influence on measurement)	Adjustment with temperature coefficients above *(2-5)*
	Temperature and humidity (influence on sample)	Temperature influences the volume resistance and humidity influences the surface leakage current
yes	Time interval between measurements and duration of charging	Dielectric absorption current above *(2-15)* with effects on interfacial polarization and volume charge, etc.).
	Voltage applied and polarity Voltage:	Surface leakage current and volume resistance Polarity: Electrolysis and ion migration
yes	Arrangement of the sample	Electrostatic induction
yes	Design of the measuring circuit	Creepage current, change in the position of the sample changes distance to electrode

The main point is the need to describe the method of insulation resistance measurement at evaluated temperature in detail. For an individual examination of the components of the measuring currents fixed framework conditions must be created. This is mandatory for a precise determination of the TI, by means of Arrhenius similarity, which is carried out on the basis of the data from the insulation resistance measurement.

4 Adaption of Research

Factors to be investigated are processed in this section after the adjustment formula has been improved.

4.1 Numerical modelling

Based on the adjustment function *(2-16)* an optimized adjustment function is developed.

$$t_N = t_R + R^k \cdot t_E$$

t_N converges towards t_R.

Assumption: classical conditions as in the standards, diffusion constant and E_a as well. I.e. k is constant, and so is R. The right addend must be a variable. In infinity, $t_{E\%}$ must converge towards t_E. This means that the right-hand addend results in zero if t_N converges to t_R

$$t_N = t_{R\ \%} + R^k \cdot \left(t_E - t_{E\%}\right)$$

boundary value $\quad t_N = t_{R_{50\%}}$ $\quad\quad\quad\quad \rightarrow \quad R^k \cdot \left(t_E - t_{E\,50\%}\right) = 0$

$$t_E = t_{E\,50\%}$$

Assumption: that R^k is comparable to a temperature coefficient and $t_E \cdot R^k$ converges towards $t_{R_{50\%}}$

$$t_{R_{50\%}} = t_E \cdot R^k$$

This results in a derivation for the determination of both k and R, since both express the ratio of the resistance of the same material at different temperatures, they are associated with each other. Furthermore, according to *(2-5)* the temperature coefficient supposed to be exponential.

$$t_N = t_{R\%} + R^k \cdot \left(t_E - t_{E\%}\right)$$
$$t_N = t_{R_{50\%}}$$
$$t_{R_{50\%}} = t_E \cdot R^k$$
$$t_E \cdot R^k = t_{R\%} + R^k \cdot \left(t_E - t_{E\%}\right)$$
$$0 = t_{R\%} + R^k \cdot \left(-t_{E\%}\right)$$

This leads to:

$$R = \frac{\left(t_N - t_{R\%}\right)^{1/k}}{\left(t_E - t_{E\%}\right)}$$

For R this can be solved analytically with variables for the extension factor or empirical by compression of $t_{E\%}$ and $t_{R\%}$ at different temperatures

(4-1)

$$k = \frac{\ln \dfrac{\left(t_N - t_{R\%}\right)}{\left(t_E - t_{E\%}\right)}}{\ln R}$$

According to the given example in the book (Rudorf 2016a, p. 61), the graphs in the figure are represented by these values:

significant resistance value of the reference value at t_nom	90%	80%	70%	60%	50%
Temperature					
20	4	25	123.2	667.7	3618.5
70	0.5	1	2	4	14

At 20 °C only the values up to 80% are known, the further values are expected to have a projected growth, which is exponential.

Assumption for exponential growth:
$$t = 0{,}774 e^{0{,}169\,x}$$

10	4.0
20	25.0
30	123.2
40	667.7
50	3618.5

In Figure 34 the values are plotted and a trend line with the corresponding formula
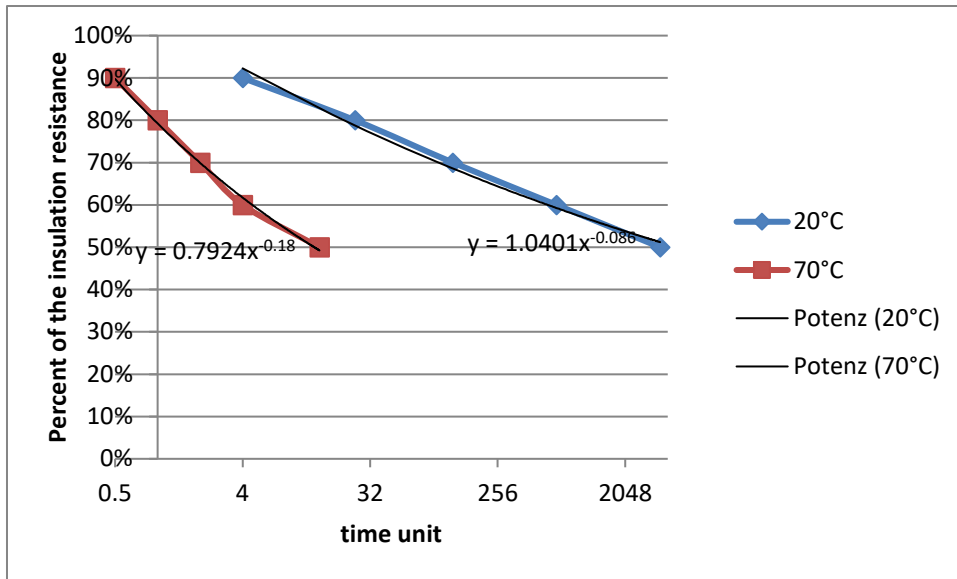
Figure 34 Example comparison baseline 20 °C - duration at 70 °C

The functions are compared, and this leads to the following solutions for R:

$$0 = t_{R\ \%} + R^k \cdot \left(-t_{E\%}\right)$$

$$R = \left(\frac{t_{R\%}}{t_{E\%}}\right)^{1/k}$$

t_E_%   0.792x$^{-0.18}$
t_R_%   1.040x$^{-0.08}$

$$R = \left(\frac{1.040\mathrm{x}^{-0.08}}{0.792\mathrm{x}^{-0.18}}\right)^{\frac{1}{k}}$$
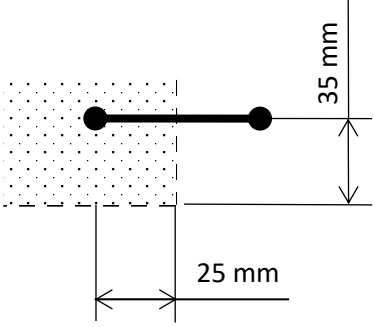
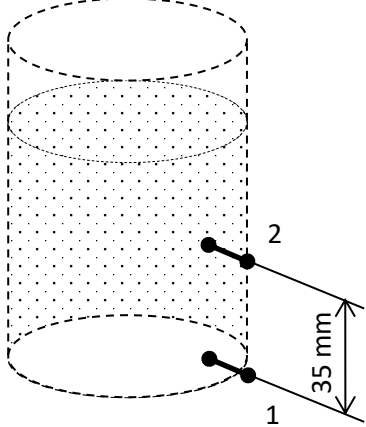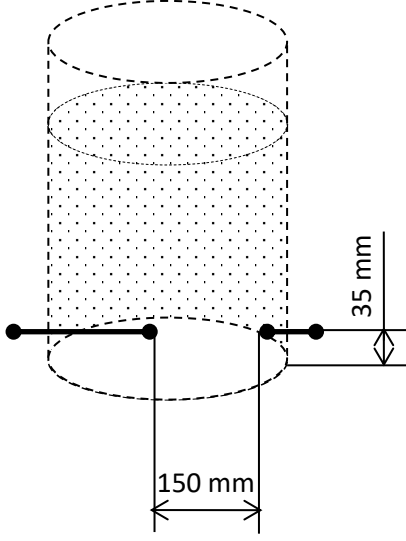$$R = \left(\frac{130x^{1/10}}{99}\right)^{\frac{1}{k}}$$

Continuing this approach with a series of tests at a different temperature would result in another function for R and k and, assuming that R is a material constant and k is the temperature coefficient, both could be determined.

4.2 Experiments

To define the relevant factors some experiments will be performed. Basically all experiments start with the same basic set-up.

4.2.1   Denotation, Coordinate system, Point of Origin

The bottom of the container is the reference level

	A stainless steel pin is inserted into the container at 35 mm above the floor. This is 50 mm long and reaches 25 mm into the container. A socket is provided at the end outside the container for connection to the meter. For simpler designation, this connection is called a water electrode.
	Optionally, some containers have a second connection in 35 mm. This is called electrode 2
	Another arrangement is an opposite stainless steel pin on the same axis. The distance between the two ends, facing each other, is 150 mm.

4.2.2 Aim and purpose of the experiments

The test programs are intended to check whether different parameters of the cables in the water bath influence the measurement result.

Relevant standards are combined as described in the past section. Although, it is not appropriate to perform experiments, according to different standards and then compare the measurement results. Different cable lengths are specified in the respective standards and the dimensions with which the cable is rolled up as a ring or wound around a cylinder are sometimes not specified or not uniform.

In addition, artificial ageing is usually carried out in the air-conditioning oven and only for measuring the cable sheathing does the insertion take place in water. It must be assumed that the positioning of the cable and the measuring electrode does not remain identical during iteration of measurement and aging.

4.2.3 Test program

Pure water is usually used as the storage medium. However, in previous experiments by Koldrack and Erfurth (Koldrack 2009; Erfurth 2016) it was pointed out, that pure water results in an altered hydrolysis and the use of artificial seawater is preferable, since more realistic results were achieved.

The program uses artificial seawater according to the DIN 50905-4 standard as an storage medium. It is measured with direct current, as usually for insulation resistance measurements.

In order to achieve transferable measured values between the different arrangements, measurements should be started one hour after storage according to the standard for power cables (IEC 60092-350, p. 22) and measured repeatedly at intervals of four hours (ASTM D 3032, p. 3). The orientation of the dielectric caused by the measurement can therefore be regarded as negligible and the measurement results can be compared with regard to the arrangement.

In order to reduce further sources of error with regard to the resistance measurement, an artificial acceleration of the tests by increasing the temperature is dispensed with.

4.2.4 Test time

The test time was limited to 14 days in accordance with the standard for determining the water absorption during artificial aging. (DIN EN 60811-402, pp. 6-8)

Results from Erfurth (Erfurth and Rudorf 2016, p. 42) suggest a shorter induction phase and comparable measured values are expected within 10 days.

4.2.5 Model

For insulation resistance measurements, the following model can be used to differentiate between different currents.
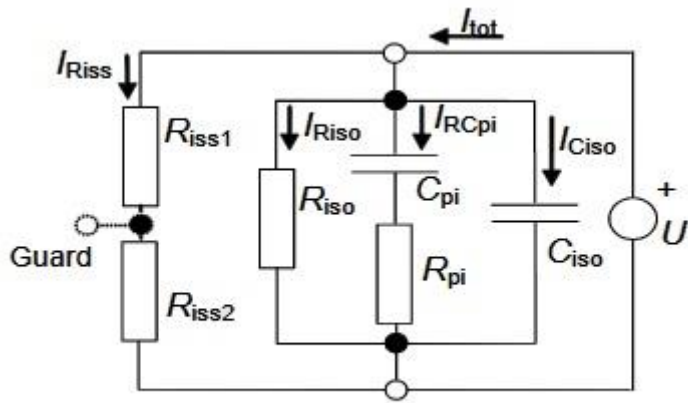
Figure 35 Equivalent circuit

U	Test voltage applied
R_{iss1}, R_{iss2}	Surface resistance
R_{iso}	Ohmic insulation resistance of the material
C_{iso}	Material capacity
R_{pi}	Polarisation Resistance
C_{pi}	Polarisation capacity
I_{tot}	Total current
I_{Riss}	Surface current
I_{Riso}	Insulation leakage current of the material
I_{RCpi}	Polarisation-dielectric absorption current
I_{Ciso}	Charge current of the capacity of the material

Guard

Surface leakage currents can falsify the measurement results. These occur from measured insulation resistances of more than 1GΩ These leakage currents flow over moisture on the surface of the insulator and are much greater than the actual leakage currents flowing through the insulator.

The guard connection was used to exclude these surface leakage currents from the measurement. This should exclude the currents flowing on the surface of the insulator from the measurement.

4.2.6   Test arrangement, devices, standards, accuracy
Cable samples:

Kabel sample „Sample"	Mass [g/m]	Measurements
Sample TPU Additive 3 27	83.8397 ± 0.0055	6.90 mm diameter, with 3 copper strands with a cross-section of 0.5 mm², the wall thickness of the insulation is 0.25 mm, braided metal shield, and the wall thickness of the material sheath 1-1.5mm

Sample TPU Referenz 23	84.0110 ± 0.0096	6.90 mm diameter, with 3 copper strands with a cross-section of 0.5 mm², the wall thickness of the insulation is 0.25 mm, braided metal shield, and the wall thickness of the material sheath 1-1.5mm

The same cable samples were used in two different cable lengths of 10.60 m and 20.60 m. Of these, 30 cm were located at both cable ends outside the water bath

4.2.6.1   Test Arrangement

Of a particular interest is the point in time for of the first measurement. Since the first measurement serves as reference value for the entire period of the experiment, the error will be permanently propagated. According to DIN EN 60216-1 the moment to capture the value should be 48 hours +/- 6 hours after immersion.

First approach was to define the change in percent to previous measurements at a time interval of approximately 4 hours between the measurements. The change (Δ) was calculated as follows:

$$\Delta = \frac{|R_k - R_{k+1}|}{R_k}$$

The specimens were immersed in a water bath at 25 °C for two days, then the temperature was elevated to 70 °C. The values were measured from two cables with polyurethane sheath with different additives.

The cables were positioned in food safe PP containers completely covered with water in the following arrangement. The ends of the cables are 30 cm outside of the water. These are led out through the cover, and the shield and one core are each connected to a conductor by flat plugs.

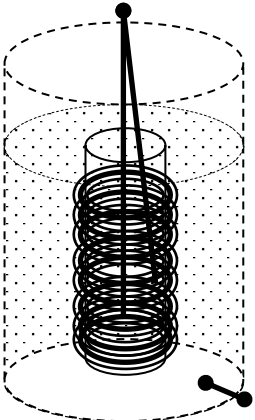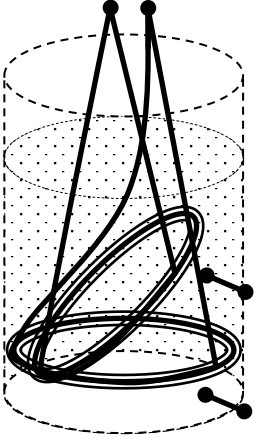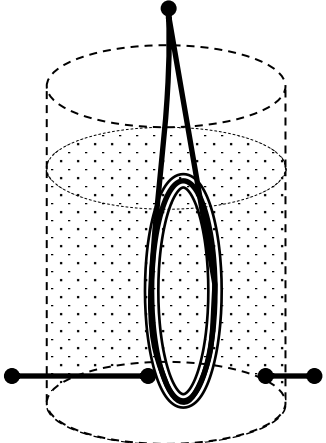| Cable #	1 10 m coiled	2 10 m coiled	3 20 m ring	4 20 m ring 45°	5 10 m ring 90°

One cable is wound tightly onto a PP pipe with an inner diameter of 100 mm, winding to winding.
The ends of the cable are guided upwards through the tube and connected outside the container.
The cylinder is lower than the water level so that water can circulate over the top and through holes at the bottom.
The distance between cable and measuring electrode is 2.5 cm.

1 / 2

Figure 36 Schematic drawing: cable coiled

 Figure 37 Schematic drawing: cable as a ring	The two cables are loosely placed in the container as a ring with an average diameter of 20 cm. The distance to the ground is 4.5 cm. The lower electrode thus has a distance to the cable ring of at least 1.5 cm The cable 2 is laid in 45° angle to the floor as a ring and has a distance to the upper electrode of 4.5 cm	3 & 4
 Figure 38 Schematic drawing: cable as a ring 90° twisted	The cable is rolled as a ring and has an average diameter of 20 cm. The ring is fixed with cable ties made of PP and is set up 90° to the floor. The distance to the short electrode is 3 cm.	5

Specification for the container:

Diameter x height (29.3 cm x 32.6 cm), volume (15.9 l), material (PP, food safe), weight (515 g), inner diameter (24.14 cm at the bottom)

When arranged as a cable ring, the 3.5 cm cables were separated from each other and from the floor by glass spacers and rolled loosely along the inner wall. As an additional variant, a 20.60 m 45° cable to the measuring electrode was installed.

For each of the two coiled cables, 10 m of cable was wound onto a PP pipe with an inner diameter of 100 mm in accordance with the following standard.

"The test pieces[...] are wound onto the cylinder (100 mm) in such a way that winding after winding rests firmly on the cylinder. Depending on the conductor cross-section, a tensile force of between 10 and 20 N may be applied". (DIN VDE 0472-502, p. 2)

Cable 1 and cable 2 were prepared with identical design for the test series, since measurement results are already available for this cable and only an adjustment between the different designs is to be carried out. Therefore, a second redundant structure was chosen for the winding.

Figure 39 cable 2 coiled on PP pipe with inner diameter 100 mm

Figure 40 cable 5 90° twisted ring with mean diameter 20 cm

<u>Circuit points</u>

"The cable to be tested shall be connected to the test equipment as follows:

Inner conductor to outer conductor, outer conductor grounded, except for armoured cables." (DIN EN 50289-1-4, p. 5)

A further adjustment was made here. After the inner conductor was measured against the screen and the absorption current was increased by reversal of polarity during the subsequent measurement, the pin sequence was reversed. Since the absorption current decreases very slowly, the dielectric was less affected by this than by a reversal of the polarity of the measuring current.

- H:S
- H:1

(the first mentioned is connected to the negative pole)

4.2.6.2   Measurement device

Insulation tester	Megger S1-1068	Nominal test voltage ±10 at 1G Ohm load Precision at 23 °C 5% to 100 G Ohm 20% up to 1 T Ohm Capacity range 10 nF to 25uF Accuracy of capacity ± 10 Meets the requirements of IEC61326-1
Multimeter	UT 71C	Input impedance up to 400 mV: >2500 MΩ, from 4 V range: 10 MΩ max. measuring voltage 1000 VDC/AC Range: 4 V Resolution: 0.0001 V Accuracy: ±(0.05%+5Digit) Range: 4 MΩ Resolution: 0.0001 MΩ Accuracy: ±(1%+40Digit)

A comparison of the expected resistances for polyurethane shows that in the area of specific surface resistances a measurement inaccuracy of 20 % can be expected from the measuring instrument and with the specific volume resistance expected errors of up to 20 % can be expected. The measurements were carried out in battery mode, so that mains fluctuations had no influence at all.

4.2.6.3   Artificial seawater

Artificial seawater, which was produced as described, was used as the inlay medium:

28 g NaCl (A)

5g $MgCl_2 * 6H_2O$ (A)

2.4g $CaCl_2 * 6H_2O$ (A)

7g $MgSO_4 * 7H_2O$ (B)

0.20g $NaHCO_3$ (B)

Dissolve salts (A) in 885 ml of desalinated water, the salts marked (B) in 100 ml. Pour solution (B) into solution (A) in a thin stream. After resting for one day, filter off and adjust to pH 7-8 with sodium hydroxide solution. (DIN 50905-4)

The volume ratio of cable to water in the containers is neglected. When examining the arrangement, the chemical reaction is classified as irrelevant and the cables must be evenly covered with water in a comparable arrangement.

4.2.7   Execution of the test
Resistance measurement before the start of the test

The cables were measured dry in their final position in the containers.

Socket and push-in sleeve   0.6 - 0.7 Ohm

coiled cable   core, shield with flat plug and socket 0.7 - 0.8 Ohm

10 m ring   core, shield with flat plug and socket 0.7 - 0.8 Ohm

20 m ring   core, shield with flat plug and socket 0.8 - 0.9 Ohm

Electrode in water bath   Stainless steel mandrel and bush 0.6 - 0.7 Ohm

Connection to device   Cable and connections 0.4- 0.5 Ohm

As already shown in the model, these resistances can be neglected in the further measurements, since the expected measurement results shall 7 times to the power of ten.

The last inflation of the containers with artificial sea water began on 13.04.2018 at 12:15 pm. Until the time of use, the water mixture was sealed airtight, correspondingly a low CO_2 concentration diffused into the water and the pH value was between 6.5 and 7.

Time of storage Cable #	12:15 1 10 m coiled	12:20 2 10 m Coiled	12:30 3 20 m ring	4 20 m ring	13:00 5 10 m Ring
Cable mass[g]	1105	1100	1731	1733	893
Volume[m³]	0.00037	0.00037	0.00075	0.00075	0.00037
H2O[kg]	12.3	12.3	14.9		9.6
Volume[m³]	0.01197	0.01197	0.01450		0.00934
Fraction of cable/water	3.1%	3.1%	10.3%		4.0%

Starting at 12:15pm, the voltage and resistance of the water between two electrodes were measured cyclically and parallel to the insulation resistances.
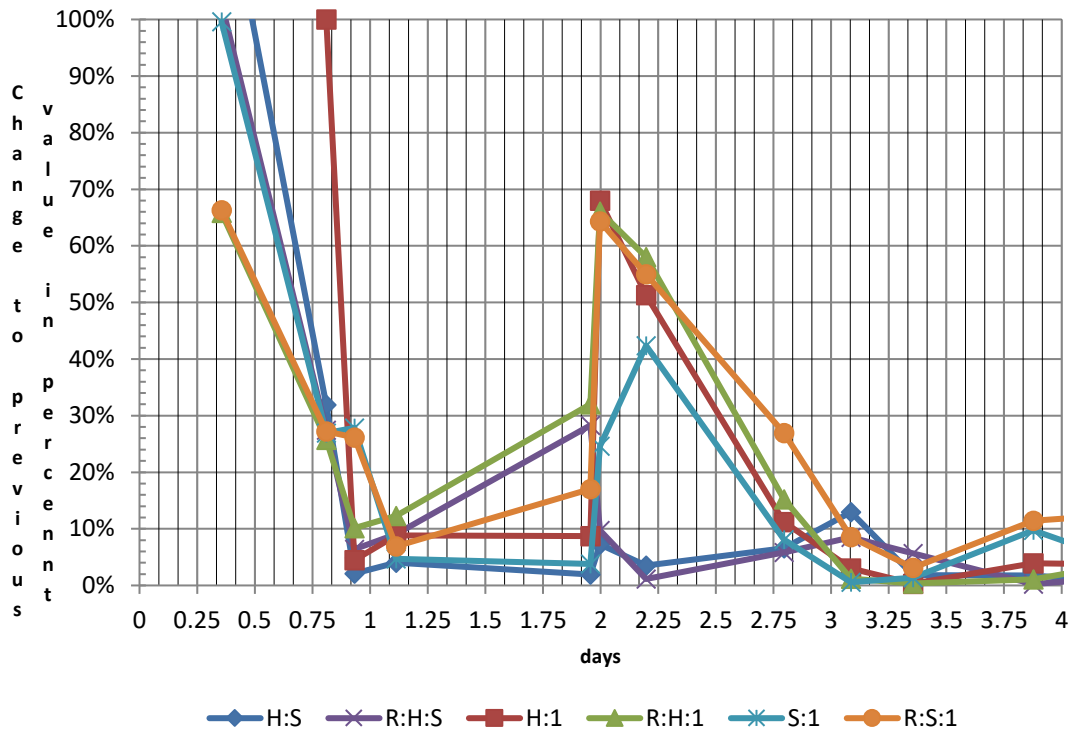
4.2.8 Point in time for the reference value

Figure 41 Change in percent -without pre-treatment

As one can see in Figure 41 the changes reach a steady level, with changes below 20 % after approximately total 66 hours.

A second experiment was performed were the specimen received two pre-treatments. At first the specimens were immersed in a water bath for 3 days at 70 °C, then the specimens hung dry in an oven for 3 days at an elevated temperature of 65 °C. In one case, for specimen number one, the air in the oven was changed continuously. After the pre-treatment the specimens were put in a water bath at an elevated temperature of 70 °C again. As one can see in Figure 42 the cable which was in the oven with continuous exchange of the air reaches steady values later than the other two. This is related to the exchange of air. Specimen number one was able to dry, due to decreasing humidity in the oven.
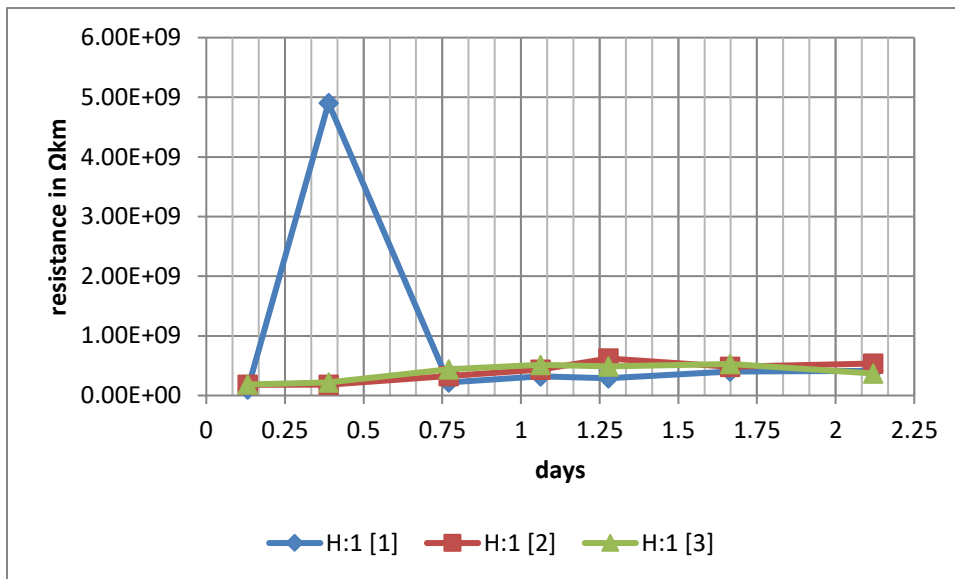
Figure 42 Change after pre-treatment

A third experiment was performed were the specimens received no pre-treatment and were the resistance was measured at 25 °C. As one can see the values reach a steady mode after only 36 hours.
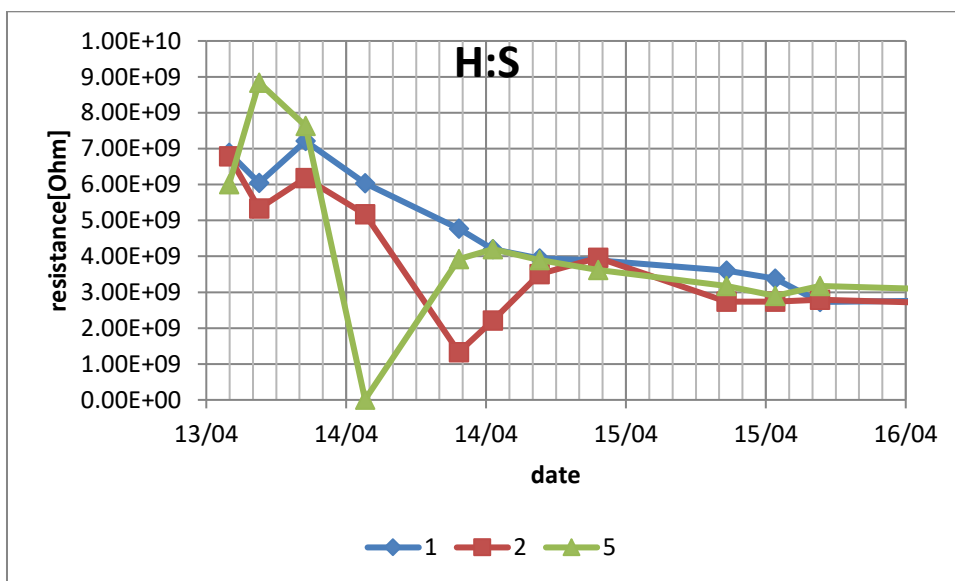

Figure 43 Change at 25 °C

To conclude, one can see temperature changes are having a certain influence and the pre-treatment has an influence, too. When the temperature is constant the values reach a steady mode after approximately 36 hours, with or without pre-treatment. For the pre-treatment it is important whether it has an influence on the moisture level of the sample.

4.2.9 Mutual influence of consecutive measurements

The time boundary between two measurements is not specified in the standards. The instructions of the measuring instrument manufacturers recommend minimum measuring intervals of 30 minutes (Chauvin Arnoux, p. 8). There are two reasons for the time period between measurements in water. On the one hand, the dielectric of the insulation material requires a certain relaxation time and on the other hand, measurements in salt water are based on electrolysis.

An experiment was performed were the resistances of one cable were measured with increasing time interval and alternating second contact.
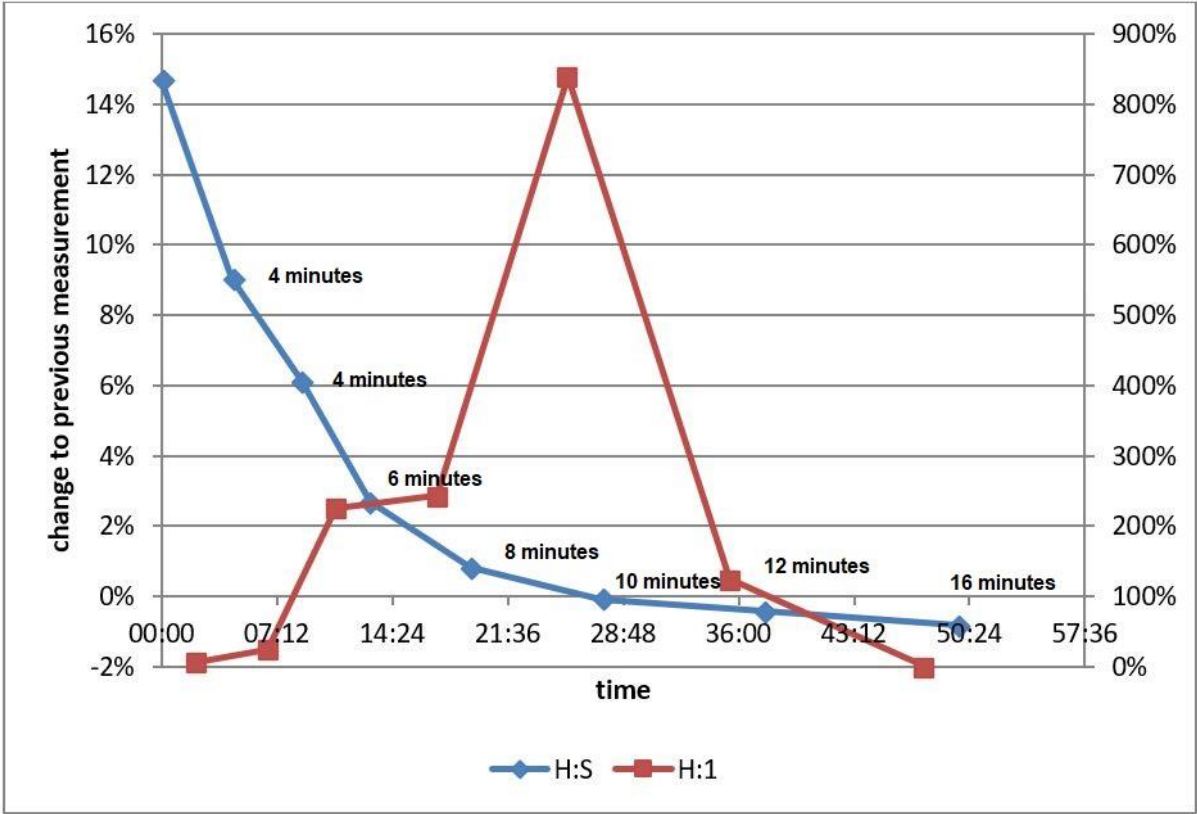

Figure 44 Mutual influence of consecutive measurements

As one can see in Figure 44, for the values of the shield (H:S) the changes decrease over time and the values actually reach negative ranges, when the interval exceeds a certain number of minutes. This can be explained with the relaxation time of the dielectric. The insulating material is charged over approximately 28 minutes and then the polarization is decreasing again. The previous time interval between two measurements was 8 minutes and then 12 minutes. This means that the insulation material with a polarization at the level it has reached after 28 minutes requires energy in less than 10-minute intervals to remain aligned. With the time of 28 minutes the polarization has reached a higher level than after the first measurements, which is indicated by the size of the changes. During the first measurements, the absorption current of the insulation material decreases faster than during the measurement from minute 14. The absorption current is decreasing slower to the end, this means the relaxation time is minimum 10 minutes with a polarization time of about 28 minutes.

A second experiment was realised at ambient temperature (25 °C) the storage water (artificial seawater produce according to ASTM 1143-98) was examined after an insulation resistance measurement.
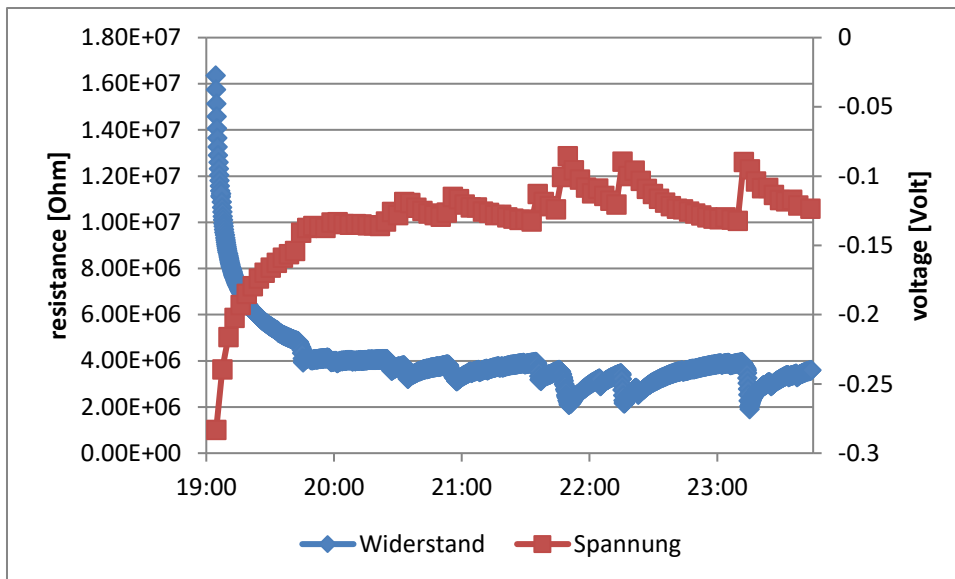
Figure 45 resistance and potential of the artificial seawater

Figure 45 shows the change of water right after an insulation resistance measurement with 500V over 2 minutes. The potential then decreases for about an hour and a half. The measured insulation resistance between two stainless steel electrodes, which were 15 cm apart, drops to a value of 4 megohm within a good hour. The noise can be interpreted as bubble formation at the electrodes caused by water electrolysis.
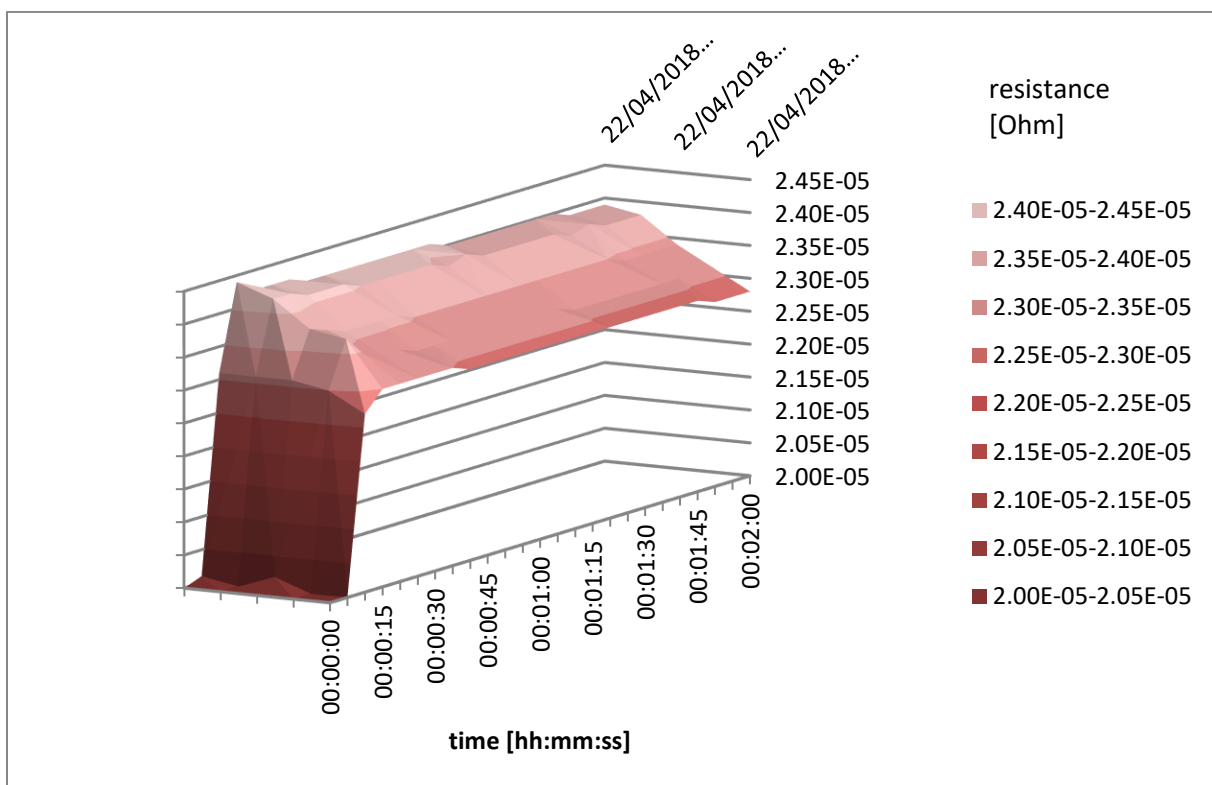

Figure 46 Polarisation

When measuring the insulation resistance at intervals of less than 10 minutes, the measured currents shift because the dielectric does not have sufficient time to relax and the absorption current increasingly drops, which influences total resistance, as shown in Figure 46 from left to right.

To conclude, the intensity of polarization of the material depends on the material itself. The process of electrolysis of the artificial seawater is initiated by the measurement. The second process lasts longer and thus defines the time interval between two consecutive measurements. For the application of insulations resistance measurements in artificial seawater the time interval should be minimum one hour.

4.2.10 Temperature coefficient

The temperature fluctuations of the measured values are limited by the respective standard to between ±2 °C in ASTM D 3032 and ± 5°C in DIN 60216. For insulation materials the specific resistance increases within a certain temperature interval, thus their temperature coefficient decreases, it is called negative temperature coefficient (NTC). For example two standard polyurethane cables with given specification (Gallagher Corporation) have a NPC, see Figure 47. Under these conditions, their temperature coefficient changes linearly with temperature, but is constant over time.

| Material | change of temperature coefficient | | |
	absolute for 46 °C	per 1 °C	percent per 1 °C
GC1090	-0.009	-0.0002	0.07%
GC1095	-0.049	-0.0011	0.25%

This results in a deviation of the temperature coefficient for a linear behaviour of max. 1.25 % for GC1095 within the permissible fluctuation range specified in the DIN 60216 standard.
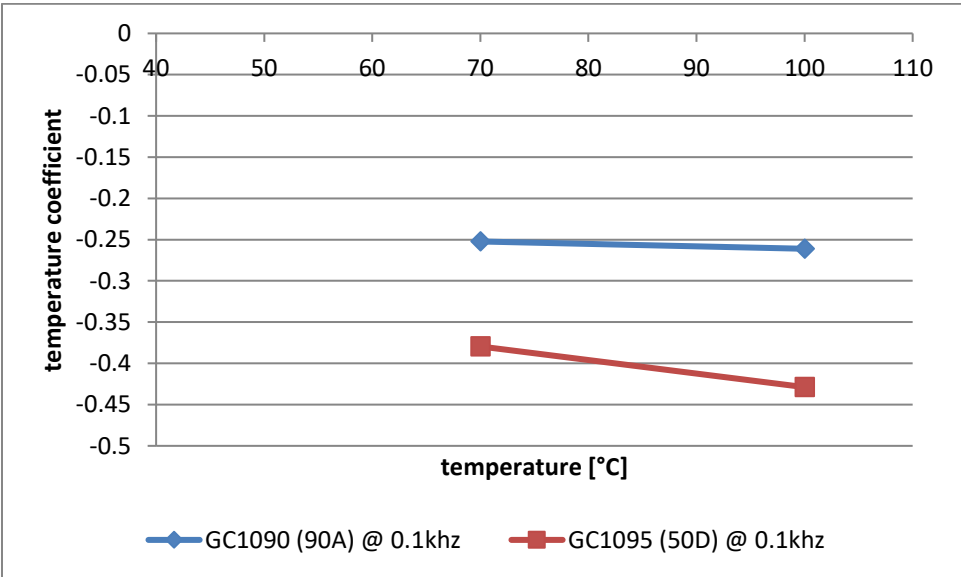

Figure 47 NPC of polyurethane

For the electrolyte one assumes an exponential temperature coefficient. Measured resistance from 19.5 °C to 80 °C result in a graph shown in Figure 48, approximated as a function:

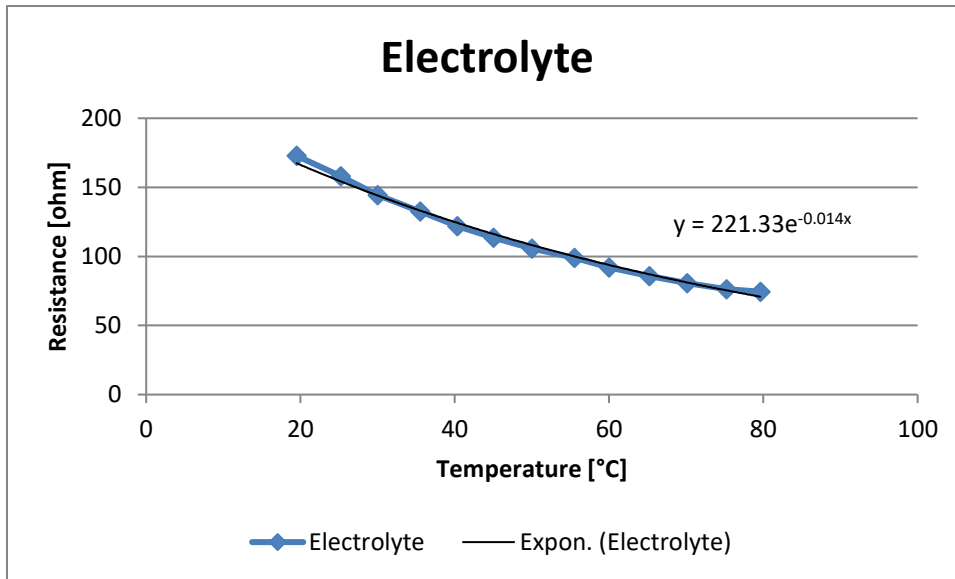$$R(T) = 227.05\Omega \cdot e^{-(T/71.42)}$$

Figure 48 Resistance of water for temperature interval (19.5 °C - 80 °C)

Taking diffusion and hydrolysis into account, the composition of materials and water changes at elevated temperatures of 70 °C and more, resulting in a temperature coefficient that changes over time. This leads to an error which is increasing over time. The best way to reduce the effects of changes over time is to convert the reference value to the elevated temperature. Thus, for each material one should determine a temperature coefficient before the experiment.

4.2.11 Test set-up

In particular the arrangement of the cables, geometry and position relative to the electrode in the water must be examined, as the standards do not provide any general recommendations. According to the law of induction, inductance is based on the temporal change of a conductor in a time-invariant magnetic field, or on the action of a temporally variable magnetic field on a static conductor. The last one is self-induction. This is caused by a temporally variable current flowing through a coil (Ose 2013, 195;200). Due to the fact, that the current is not constant, self-induction is a possibility to skew the results.
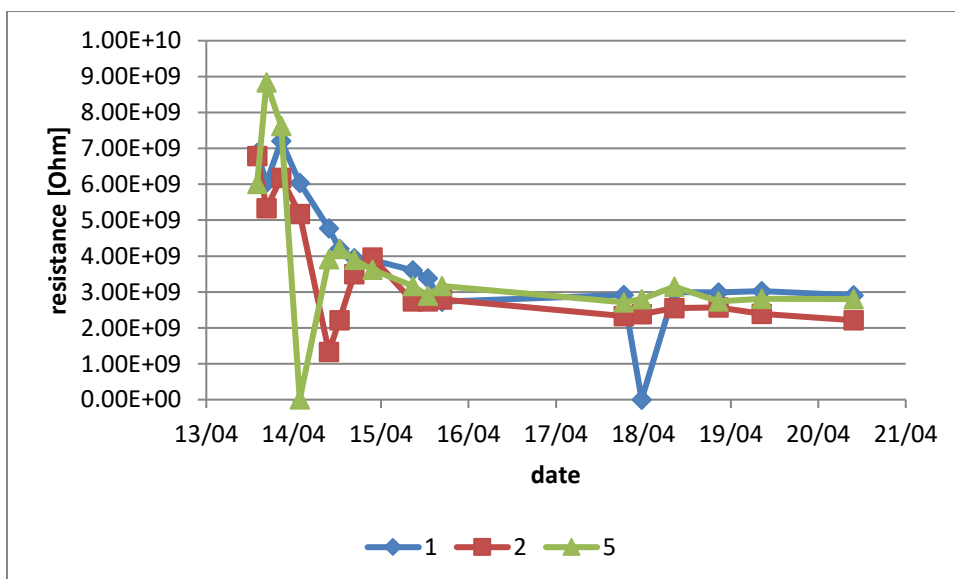

Figure 49 Coiled cables on 100 mm PP pipe

The cables number one and two in Figure 49 are coiled on a PP pipe with a diameter of 100 mm, each is 10 m long and wound layer by layer onto the PP pipe. Cable five lies loosely as a ring in the basin. The diagram shows that the measured values for eight days lie within the measuring inaccuracy of 5 %, with the exception of some outliers during the levelling during the first 36 hours and one spike at the 18th. One can say that the winding of the cable has no relevant influence on the insulation resistance measurement.

The position of the cable relative to the electrode is relevant, when measuring the insulation resistance against water, since the resistance is directly proportional to the length of the virtual conductor. As one can see in Figure 50and Figure 51, the position of the electrode has a significant influence on the result. The difference of the resistance of the sheath is almost 25 %. The insulation resistance between water and core is not so strongly affected because of a smaller fraction of the resistance of the water in the overall resistance. Thus, the influence of the virtual length of the conductor in the water is considerably less.
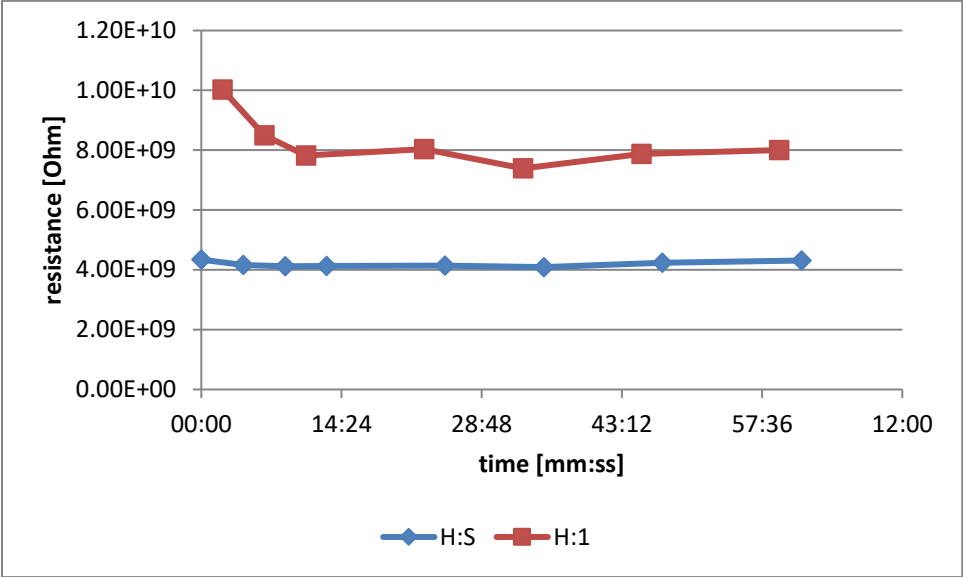

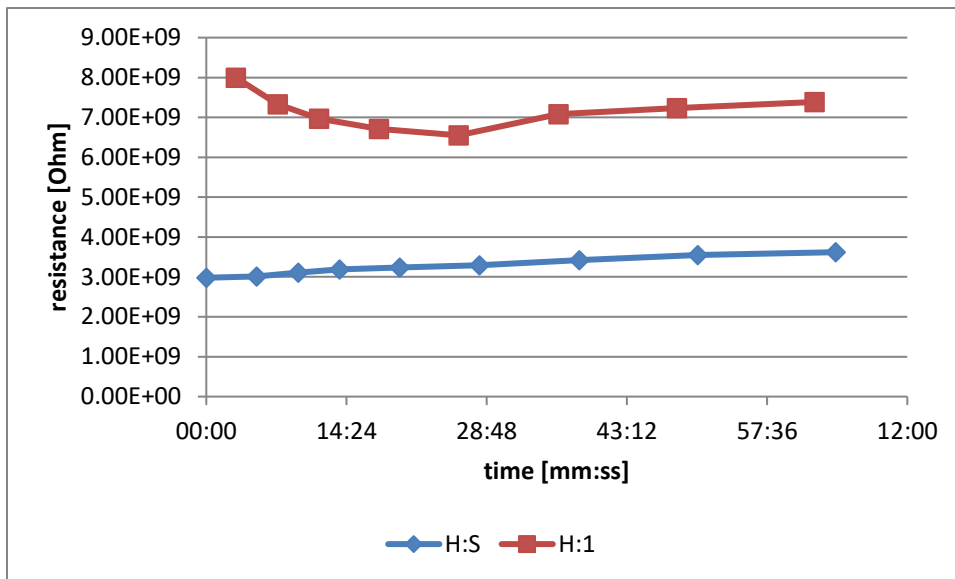*Figure 50 Long distance to electrode*

Figure 51 Short distance to electrode

An additional influencing factor is the surface leakage current. Although, the three terminal test is already explained in the section *2.2.1,* but to illustrate the order of magnitude, Figure 52 shows that the surface leakage current is three times a power of ten.
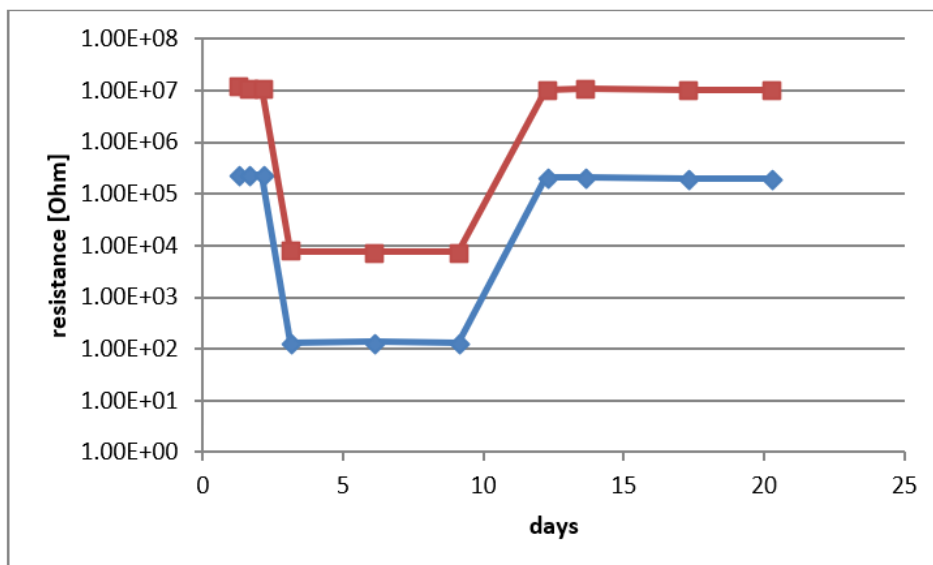

Figure 52 Surface leakage current due to missing guard connection

It is particular important to understand the different currents and to not change the set-up during a running test. As can be seen schematically in Figure 53, all currents are parallel to each other, so that the small currents cannot be neglected.
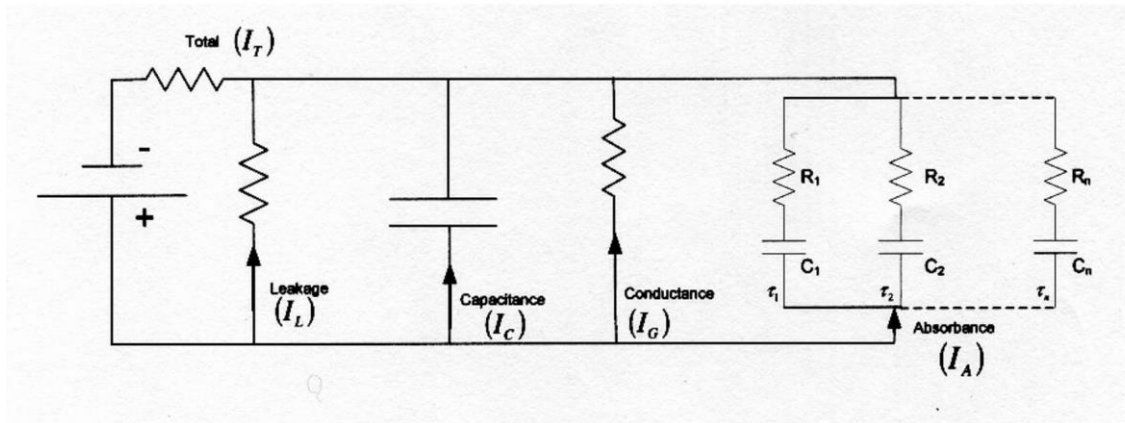
Figure 53: Equivalent circuit showing the four currents monitored during insulation resistance test (IEEE 43, p. 5)

4.2.12 Seawater composition (change over time)

The gases diffusing from the air into the water have the following calculated saturation limits in the water:

Principal constituents	Concentration	[mol]	Henry Law constant [atm/mol]		saturation point [mg/l]
N_2	0,79	28,014	1,20E+05	1,91E-04	5,35E+00
O_2	0,21	31,998	6,29E+04	9,68E-05	3,10E+00
CO_2	0,04	44,008	3,41E+03	3,40E-04	1,50E+01

Temperature
70°C

1 litre saltwater   34,482

As shown in Figure 54 with increasing temperature at a constant pressure of 1 atm the solubility is decreasing.
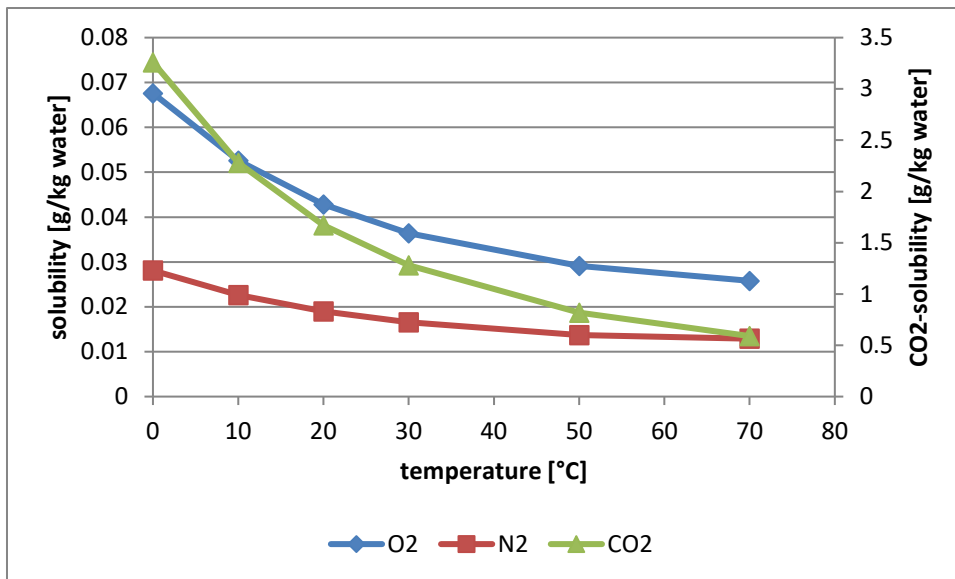
Figure 54 Solubility in water

Based on the change in salinity, presented in the definition of water, and the pH change shown here, the chemical activity of water changes in correlation with the change in temperature.

As a side-effect of the insulation resistance measurements in saltwater, the applied current and the dissociated ions create an electrolysis. Without alternating current the electrode with a surface in the seawater will sacrifice due electrons. The resistance of the electrode is changing over time as shown in Figure 55. Within one week the resistance changed from $1.5*10^6$ to $3*10^6$. The long-term pattern is similar to the 22 hours presented, after insulation resistance measurements of the cable, the resistance of the electrode in water registered a high amplitude, which decreases within half an hour. Small peaks occur frequently during the rest of the time.
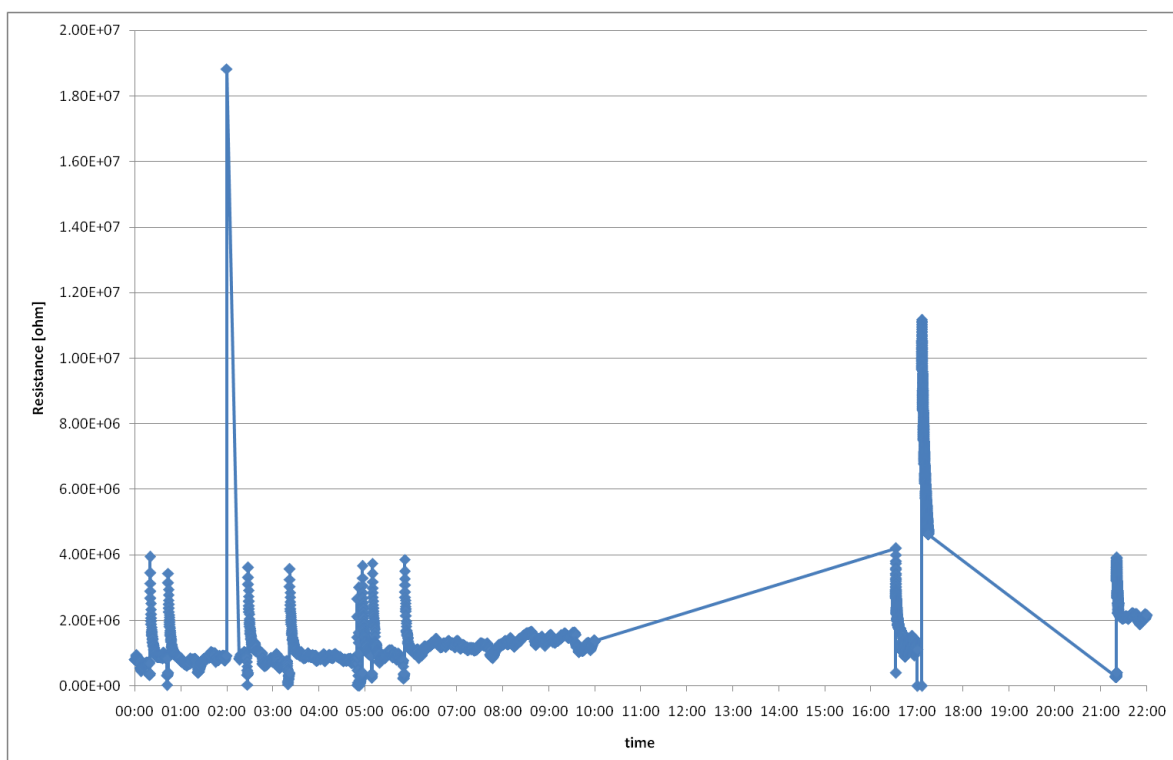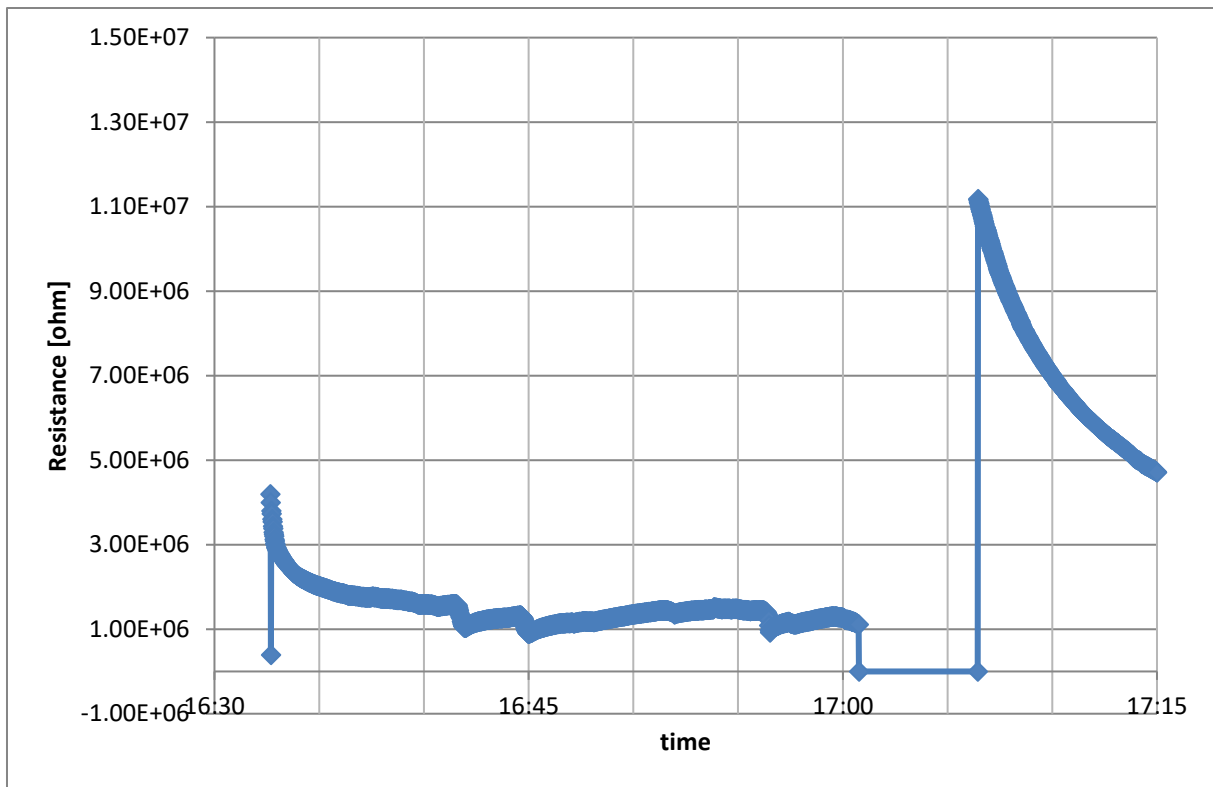
Figure 55 Resistance of water over 22 hours

Figure 56 Water resistance after measurement

A typical trend that represents the change in the curve after a measurement is shown in Figure 56. One can see that the resistance is increasing very briefly for one order of magnitude to the power of ten. Thus, the electrical field, which is applied must have some effect on the water.

4.3 Evaluation and corrections

Priority error calculation for the accuracy of the length for the cable lengths used.

Calculation of errors:

Mean: $\bar{x} = \sum_{i=1}^{n} x_i$ (2)

Standard deviation   : $\sigma = \sqrt{\dfrac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$ (3)

Confidence Interval: $\bar{x} \pm t \cdot \dfrac{\sigma}{\sqrt{n}}$ (4)

With Confidence level 90% $t_{90\%} = 2.92$

Gaussian law of reproduction:

The weight per meter (z) depends on the measurement result for the two directly measured quantities weight (x) and length (y).

Mean : $$\bar{z} = f(\bar{x}; \bar{y})$$ (5)

Standard deviation of the mean value (mean error of the mean value):

$$\Delta z = \sigma_z = \sqrt{(f_x(\bar{x}; \bar{y})\Delta x)^2 + (f_y(\bar{x}; \bar{y})\Delta y)^2}$$ (6)

Measuring result: $$\bar{z} = \bar{z} \pm \Delta z$$ (7)

The confidence level in the weight determination has resulted from the fact that the balance has an inaccuracy of 0.1 mg. For the sample to contain the expected value, the value with maximum deviation plus measurement inaccuracy was taken and the confidence level for which the confidence interval is sufficient was calculated back.

The maximum deviation of 16774.4 + 0.1 mg was calculated from the measurement results of cable 23.
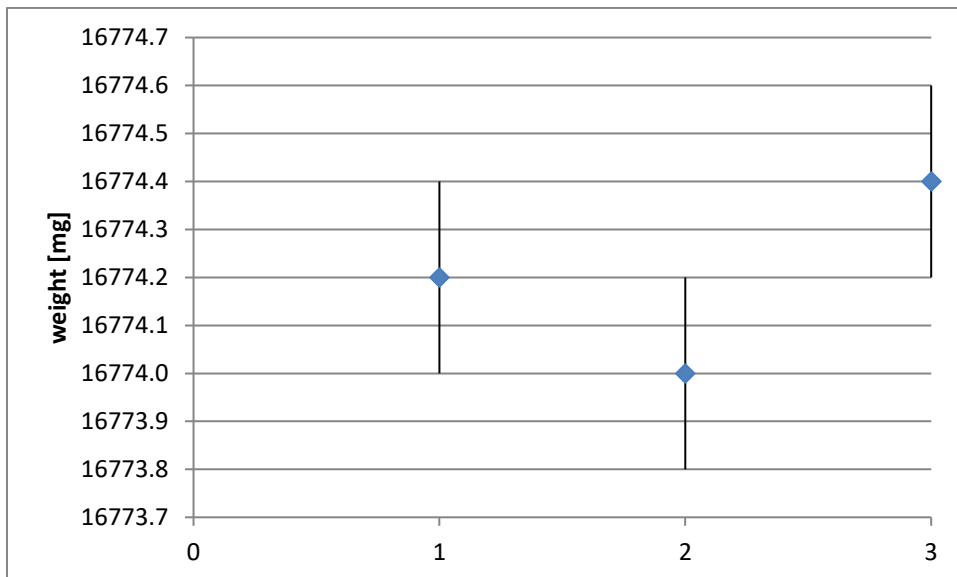

Figure 57 Measured values of the mass of cable 23 with standard deviation

The confidence interval must therefore reach an upper limit of 16774.5 mg. This results in a level of confidence of 90%.

The average error in mass per meter results from the error propagation for the measured values of cable 23:

$$m = 16774.2 \pm 0.3 \, mg \text{ und } l = 199.7 \pm 0.4 \, mm$$

$$z = z(m; l) = \frac{m}{l}$$

$$\bar{z} = z(\bar{m}; \bar{l}) = \frac{\bar{m}}{\bar{l}} \qquad \rightarrow \qquad \frac{16774.2 \, mg}{199.7 \, mm} \frac{1000}{1000} = 84.0110 \, ^g/_m$$

$$\frac{\partial z}{\partial m} = \frac{1}{l} \qquad \rightarrow \qquad \frac{\partial z}{\partial m}(\bar{m}; \bar{l}) = \frac{1}{0.1997m}$$

$$\frac{\partial z}{\partial l} = m \cdot \ln(|l|) \qquad \rightarrow \qquad \frac{\partial z}{\partial l}(\bar{m}; \bar{l}) = 16.7742\, g \cdot \ln(|0.1997|) = -27.0250\, \frac{g}{m^2}$$

$$\Delta z = \sqrt{\left(\frac{\partial z}{\partial m}\Delta m\right)^2 + \left(\frac{\partial z}{\partial l}\Delta l\right)^2} = \sqrt{\left(\frac{1}{0.1997m} \cdot 0.0003\, g\right)^2 + \left(-27.0250\, \frac{g}{m^2} \cdot 0.0004\, m\right)^2}$$

$$\Delta z = 0.0096\, \frac{g}{m}$$

Thus, the confidence interval of cable 23 for the function of the ground lies per length:

$$z = 84.0110 \pm 0.0096\, \frac{g}{m}$$

and analogy for cable 27:

$$z = 83.8397 \pm 0.0055\, \frac{g}{m}$$

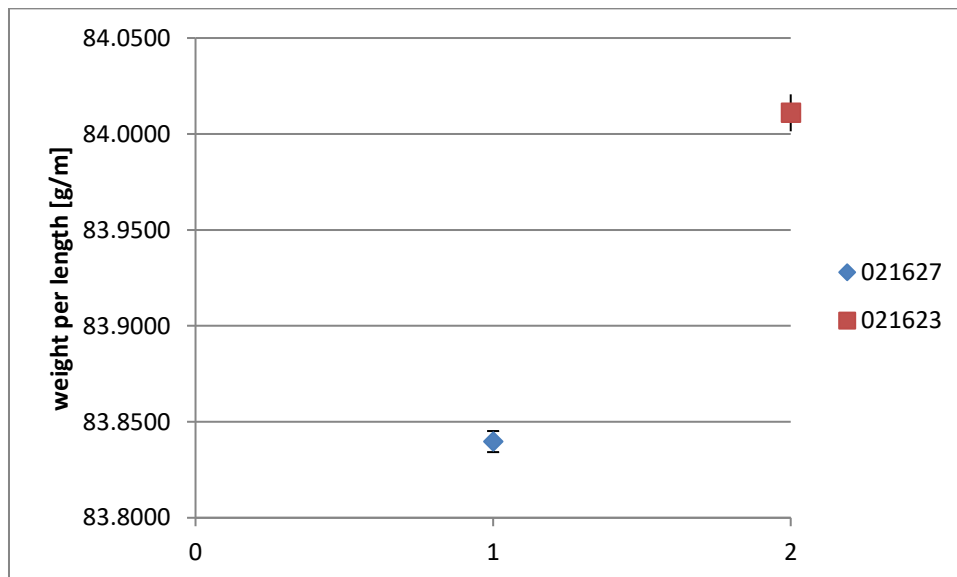*Figure 58 weight per length with confidence interval*

For the insulation resistance measurement, with a distance of four hours according to ASTM D 3032 was maintained, but the behaviour of the water was also checked by measuring the voltage between two electrodes in the water.

The times of voltage and resistance, independently measured in water, indicate relaxation of the water within approx. 40 minutes.

15.04. 17:00 before measurement
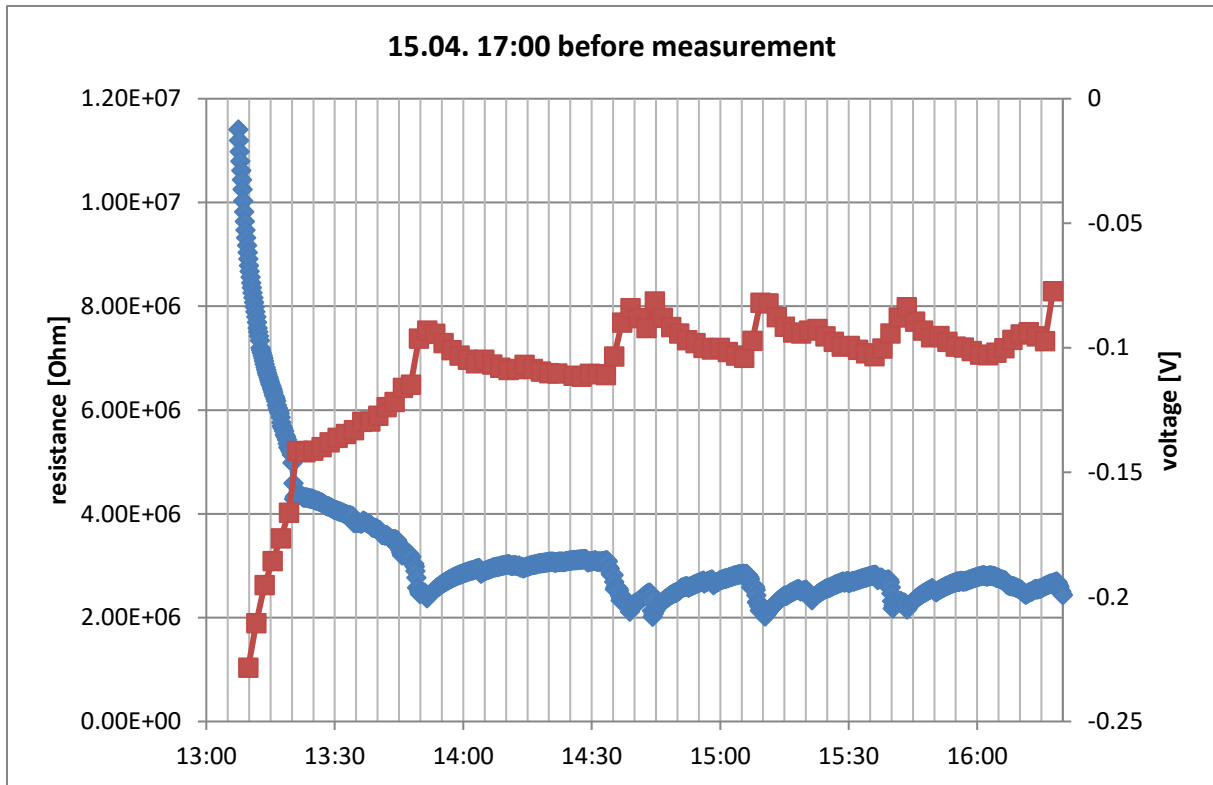
The resistance deflections indicate voltage fluctuations of the multimeter (#1 resistance), or surface changes on the electrode (electrolysis). Regardless of this, the resistance of the water continuously decreases after the insulation resistance measurement.

15.04. 17:00 after measurement

voltage drop 5 min
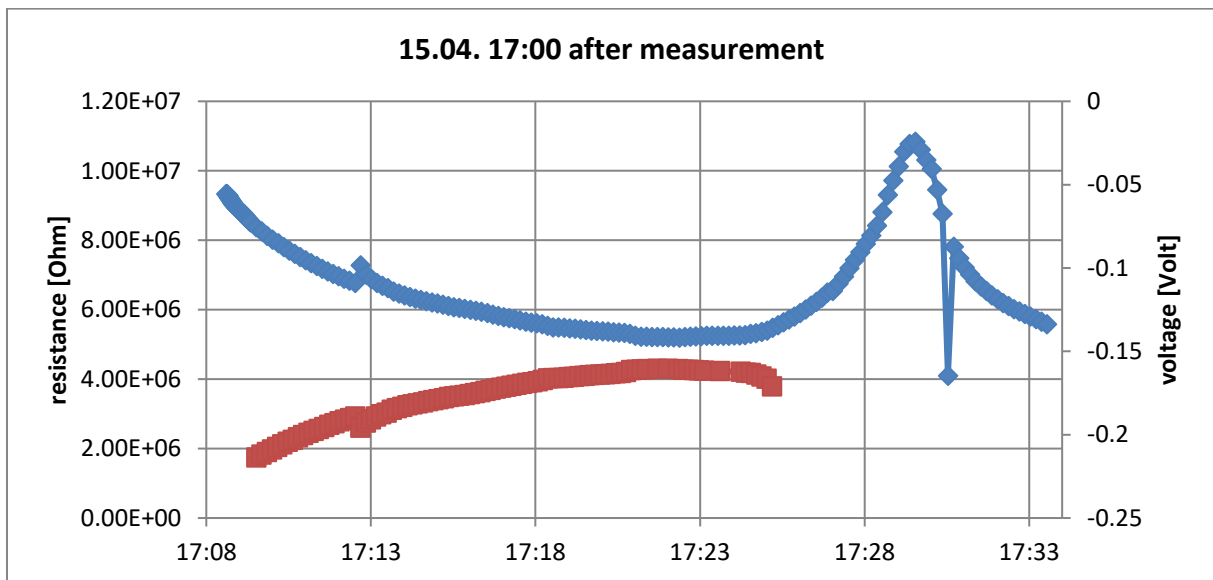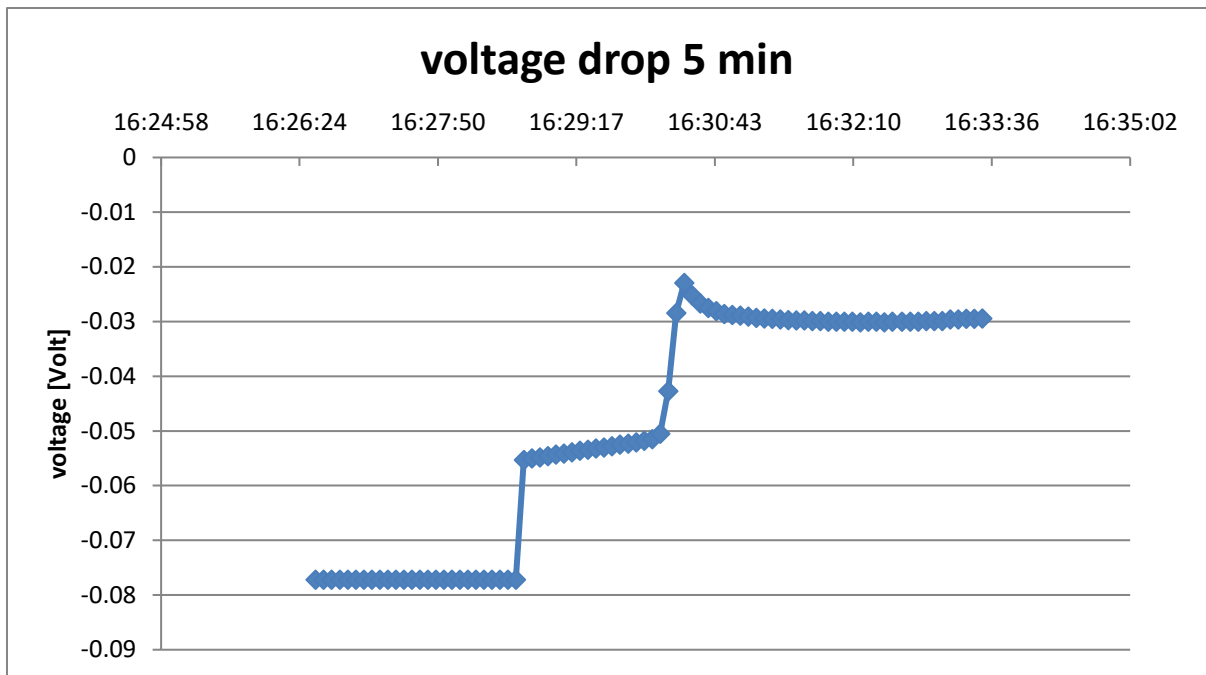
Within the first minutes after the insulation resistance measurement, the potential changes and the voltage for measuring the resistance is increasing. In addition, the voltage rise takes place after separation of the multi-meter for resistance measurement (voltage source approx. -0.171 Volt) from the circuit.

The combined resistance-voltage measurements directly after the insulation resistance measurement indicate that the compensation current initiated by the resistance measurement increases briefly.
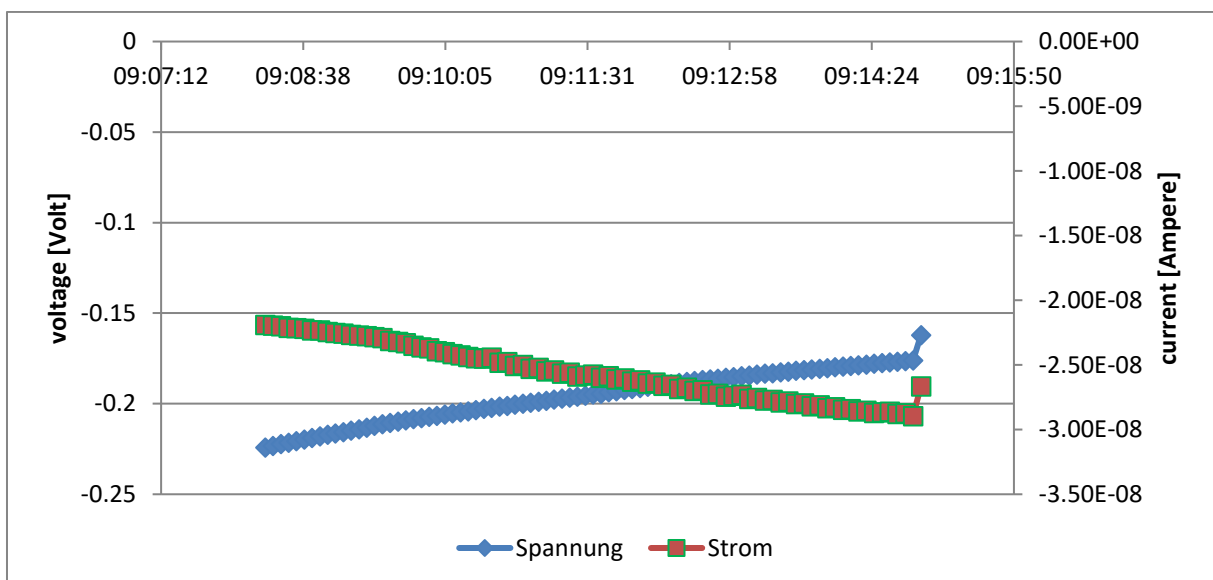
5 Discussion of the results

In this work, the condition of the insulation material was described by measuring the insulation resistance. The insulation resistance measurement has proven to be more robust compared to previous investigations and it is no longer necessary to record which synthetic fibres the tester wore.

But this does not hide the fact that the measuring error of up to 20% is caused by the measuring device itself. Newer approaches for clarifying the Arrhenius similarity have also been found in the literature. But the regulations, which define the requirements, that the cable sheath must meet are so outdated, that the prediction does not require any clarification. Besides the fact, that the failure rate is so little, that almost nobody is aware of the potential to reduce the sheath. Despite the fact, that the risk of failure is associated with very high financial loss, due to very expensive maintenance for offshore cables.

The investigated method can nevertheless be considered successful, since the application of the adjustment formula is regarded as promising and thus represents a solid manipulated variable for optimizing the prediction of the time to failure.

With regard to the system immanent influence of the water, a subsequent investigation is recommended, with the objective of a simulation. This statement is specified in the findings. The measurement itself has a decisive influence on the water, as the water tends to electrolysis due to the current applied.

The literature research has also shown that, with regard to pressure simulation, further chemical processes taking place, due to the increase in dissolved carbon. A changed salinity can be observed, too. But non the less with increasing depth the saturation rate is decreasing and this leads to a reduced diffusion. The lower temperature in deep sea reinforces this slow down. To conclude adsorption is neutral facing pressure, which means the process is neither increased, nor decreased.

With regard to the sheath material it needs to be investigated for further details, due to limited equipment and missing water analysis the influence of additives are just a guess. But standards which recommend to test each colour separately seem to underline the influencing potential of additives.

6 Summary and outlook

The first step towards a more reliable prediction of the useful life using current scientific methods is the precise recording of previously only estimated values using newer measurement methods. The determination of the activation energy with the aid of TGA and DSC or the measurement of the change in polarization using the recharging effect instead of the description of the dielectric using the insulation resistance as an indicator of the change over time can be considered. Simulation taking higher order chemical reactions of polyurethane into account is already possible in connection with the calculation of parallel Arrhenius processes. The degree of crosslinking in the simulated aging process could also be increased in comparison to the actually aging polyurethane, in order to compensate for the influence of thermal mobility on the adsorption, which is actually temperature-independent.

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Appendix

The source code which was produced for processing the large amount of data will not be include but can be enquired at any time.

I. Mindmap of standards