

ISE Primer

ION SELECTIVE MEASUREMENT IN ONLINE ANALYSIS

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Ion selective measurement in online analysis

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1 Fundamentals of the ion selective measuring technique

1.1 The principle

Analytic measuring technique provides a huge number of procedures to determine the concentrations of substances in an unknown sample, e.g. photometry, titration or mass spectrometry. One advantage of working with ion sensitive electrodes (ISE) is that it is possible to obtain meaningful data on individual substances even with economical systems. Otherwise, this is only possible with much more expensive instrumentation. Ion sensitive electrodes are electrochemical sensors that can be used directly in the mg/L range and below. The WTW developments demonstrate that this measurement principle is also suitable to continuous online operation.

Analysis with ISE is among the so-called potentiometric procedures. This means the wanted signal occurs in the form of potentials, more precisely of potential differences, or voltages in mV.

An ion sensitive measuring system basically consists of the ISE reacting on a special ion type and a reference electrode that are jointly immersed in the sample to be measured (Fig. 1).

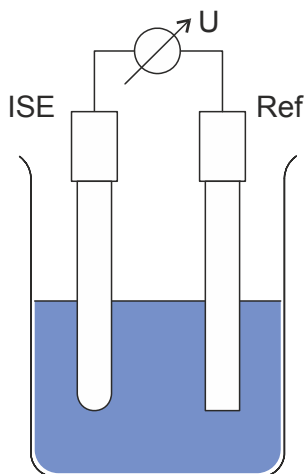


Fig. 1 Schematic structure of an ion sensitive measuring system with ISE (ion sensitive electrode), Ref (reference electrode) and voltmeter

This combination is called a measuring chain. This measuring instrument must be very highly resistive in order to fulfill the conditions of a potentiometric measurement. Only then are the measurement currents low enough so no value changing polarization occurs and the electrodes are not damaged. For these reasons the electrode connections must be kept dry. Moisture can cause unwanted parallel connections and interferences.

1.2 The Nernst equation

The ion sensitive electrode provides an electrochemical potential that is influenced by the concentration of the ion type to be measured. The reference electrode, however, is intended to build up an electrochemical potential that does not depend on the composition of the sample to be measured. The difference of these potentials, the voltage displayed by the meter, can be described by the Nernst equation (explanations in the appendix):

$$U_{ion} = U_{ion}^0 \pm S \cdot \log(a_{ion}) \quad (1)$$

U_{ion}^0 is a fixed value given by the measuring system. The slope S defines how much the measurement signal is increased or reduced when the concentration changes. Its theoretical value is referred to as the Nernst slope and is 59.16 mV at 25 °C for simply charged ions such as ammonium or nitrate. The arithmetic sign depends on the charge of the measured ions, plus is for positively charged cations, e.g. K^+ , minus for anions such as Cl^- .

The activity a is the active concentration of an ion type that takes into account the impact of other ions in the sample and thus describes a considerable part of the matrix effect. Suitable procedures for calibrating and measuring as described in this primer make it possible to work in practice with the adapted form of the Nernst equation

$$U_{ion} = U_{ion}^0 \pm S \cdot \log(\beta_{ion}) \quad (2)$$

where β is the mass concentration, for example mg/L.

As an example, a graphic of this function is shown in Fig. 2. Note that the concentration value is scaled logarithmically. The measured voltages are mostly in the range -500 mV to +500 mV.

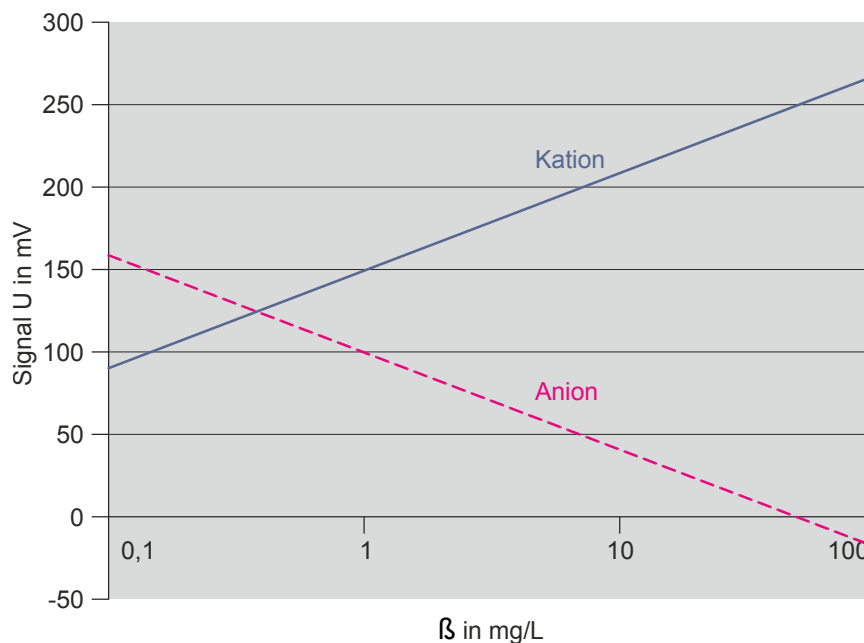


Fig. 2 Theoretical run of calibration line for anions and cations (according to Nernst equation))

2 Electrodes

2.1 Structure and function of ion sensitive electrodes

An ISE has a shaft with a built-in membrane (Fig. 3). The membrane is the essential sensing device and guarantees the sensitivity for a certain ion type. The ion specific potential develops on the membrane surface according to the Nernst equation.

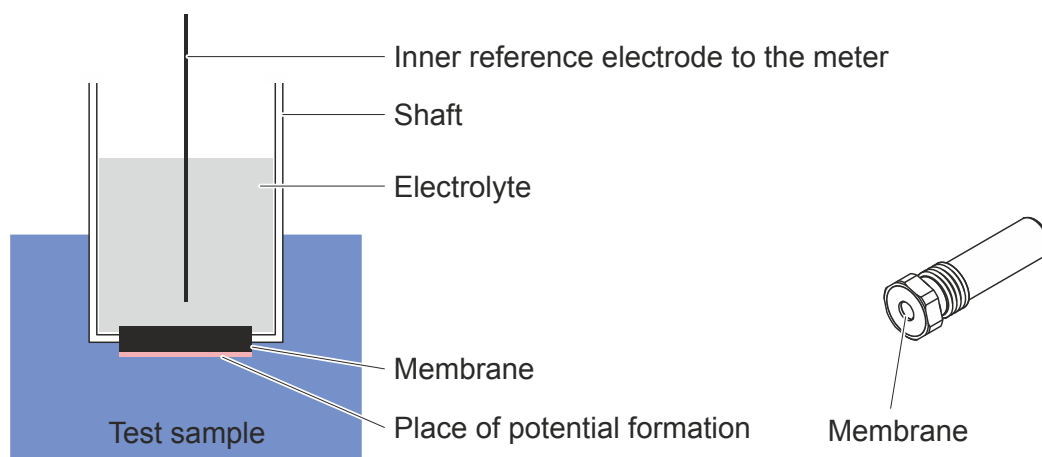


Fig. 3 Structure of an ion sensitive electrode (diagram on the left, sample model on the right)

There are various types of membranes. The most important types are glass membranes, solid body membranes and synthetic material membranes.

Glass membrane	<p>The ion selective membrane most frequently used is the glass membrane, which is used in pH measuring technique. The pH sensitivity is due to a thin expanding layer in the glass through which ions can diffuse in and out (further information, see pH primer).</p> <p>Other ions, e.g. Na^+, can also be determined with the corresponding glass and special procedures.</p>
Solid body membranes	<p>Membranes made out of hardly soluble salts such as lanthanum fluoride, silver chloride or silver sulfide can also be used to detect e.g. fluoride, chloride, sulfide or silver.</p>
Synthetic material membranes	<p>Certain synthetic materials are able to absorb ion sensitive substances. Thus, after an addition of so-called ionophores, PVC that contains plasticizers is suitable for measuring various ions.</p>

Contrary to the theoretical graphs from Fig. 2, curves in practice are often like those in Fig. 4.

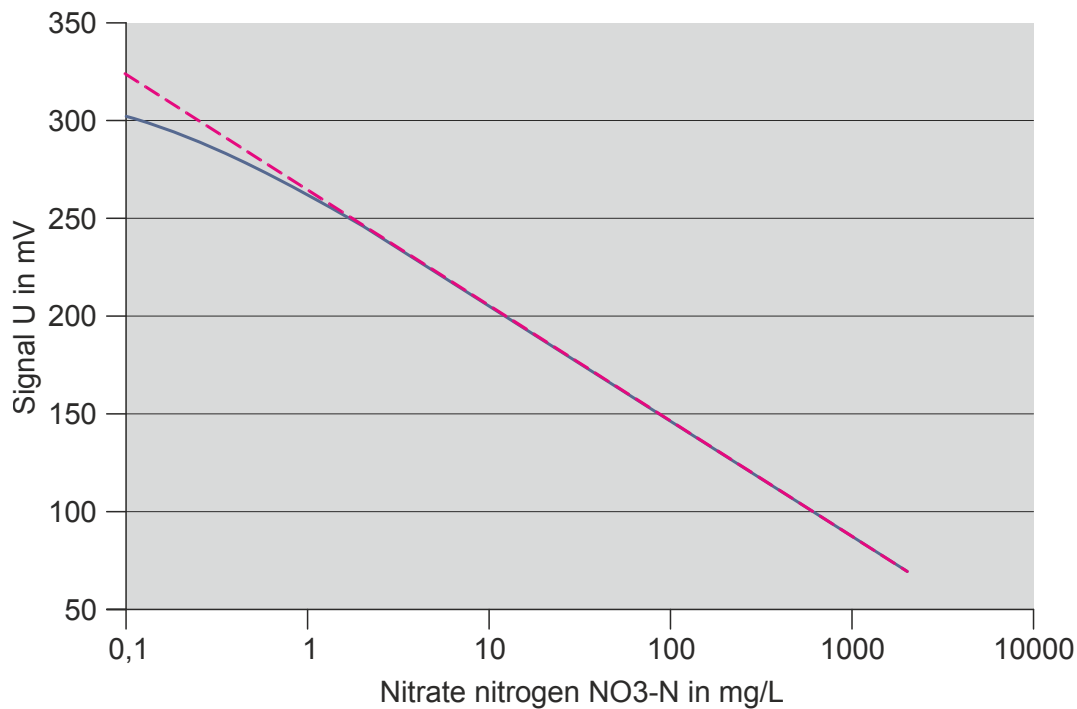


Fig. 4 Real characteristic curve of a nitrate ISE (solid line) and purely theoretical Nernst function (dashed)

This real characteristic curve differs from the straight line function in the concentration range below 1 mg/L nitrate-N. Here, the electrode reacts less strongly to the required measured parameter. In WTW measuring systems this effect is taken into account automatically. If the concentrations are lower, the value is so low the ISE no longer reacts to changes of the measured ion. This value is referred to as the detection limit and is different for individual electrode types. It also depends on the presence of other interfering ions.

For the tasks required in waste water treatment plants, electrodes for the parameters ammonium and nitrate are useful. In addition, potassium and chloride electrodes can be used for compensation purposes (see chapter 2.2). The application range of the first two parameters can thus be extended.

2.2 Impact of interfering ions

In most cases, ion sensitive electrodes react not to one ion type only but to other types as well. These are referred to as interfering ions. In the case of an ammonium sensitive electrode, potassium interferes because it acts similar chemically. The Nernst equation can be extended to describe these circumstances (see appendix). The selectivity coefficient specified there is a parameter that describes how strong the effect of the interference is. A value of $K_{\text{mess, stör}} = 0,2$ states that the interfering ion produces the same voltage as the concentration of the measured ion $\times 0,2$. If $K_{\text{mess, stör}} = 0,01$, the concentration of the interfering ion must be one hundred times higher in order to have the same effect on the measurement as the simple measured ions concentration.

When measuring ammonium with the VARiON^{®Plus} 700 IQ sensor, simple NH₄-N measurement in the presence of potassium results in the following overvalues:

Potassium contents	Ammonium value increased by approx.
10 mg/L	0.7 mg/L
50 mg/L	3.4 mg/L

This influence can be balanced by compensation. For this the interfering ions concentration has to be measured as well and to be included in the evaluation of measurement data. VARiON^{®Plus} 700 IQ provides this option. Fig. 5 shows the interrelationship of real and displayed NH₄-N value for different potassium contents. The characteristic curve with the optimum potassium compensation corresponds to the characteristic curve without any potassium content.

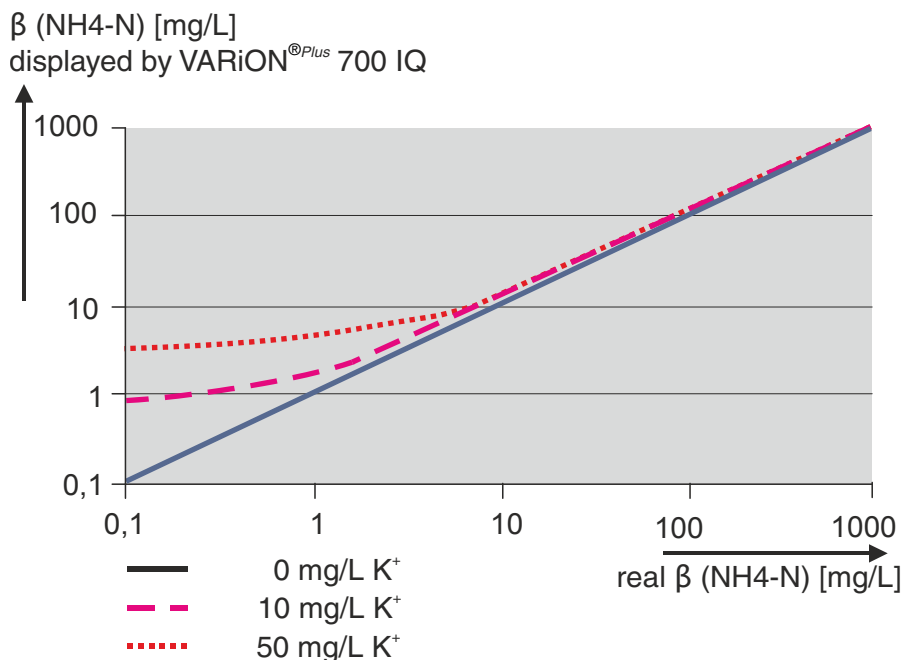


Fig. 5 Influence of potassium on the ammonium value

Whether there is a considerable interference basically depends on the ratio of measured ions and interfering ions and has to be estimated in each individual case. A concentration of 20 mg/L

potassium is important in the presence of 3 mg/L ammonium but irrelevant in the presence of 100 mg/L ammonium.

The same applies to the interference of chloride for nitrate measurement with the VARiON^{®Plus} 700 IQ sensor:

Chloride contents	Nitrate value increased by approx.
100 mg/L	0.7 mg/L
500 mg/L	3.6 mg/L

The relevant curve is shown in Fig. 6.

β (NO₃-N) [mg/L]

displayed by VARiON^{®Plus} 700 IQ

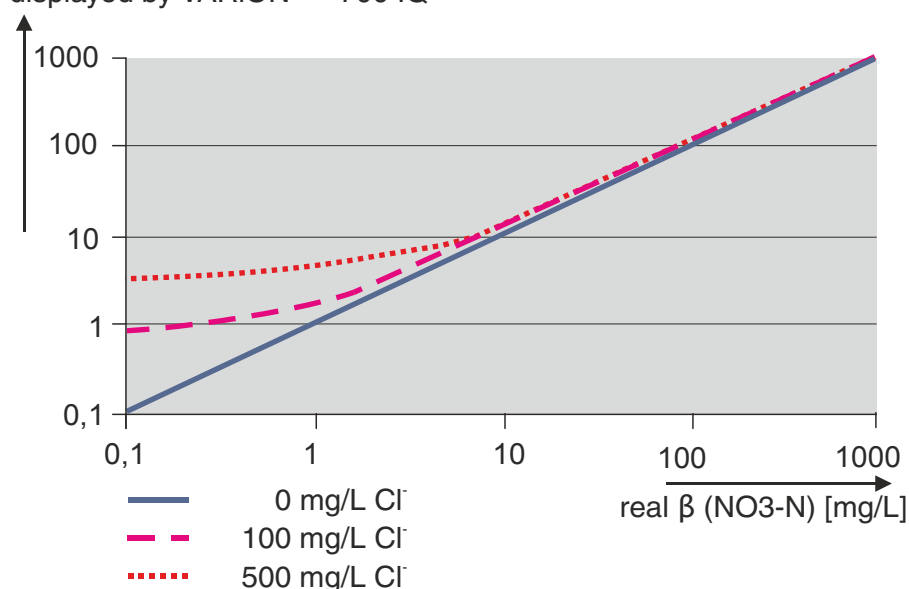


Fig. 6 Influence of chloride on the measured nitrate value

If the concentration of the interfering ion is known its influence on the actual measured ion can be taken into account. This should always be done if the uncompensated measured value is influenced beyond the required precision tolerance by the presence of the interfering ion.

Manual compensation is possible if the conditions in the measurement application change slowly or inconsiderably only. Automatic compensation is recommended if the changes are quick and considerable. Manual means that in each calibration cycle the interfering ions concentration is entered manually on the measuring system by the user. Automatic means that the interfering ion is permanently determined by the measuring system by means of an extra electrode and that the current value is included in the signal evaluation.

Measurement of the interfering ions potassium and chloride with ISE is also affected by other ions (ammonium or nitrate). The effect of this interference on the determination of the actual measured parameter, however, can be disregarded due to the selectivity coefficients.

Some parameters have special features caused by their chemical characteristics that have nothing to do with the determination process of the electrode. The pH value, for example, affects the balance between the ammonium displayed by the electrode and ammonia, which is not measured (see chapter 6.3). If necessary, this influence can be compensated for by measuring the pH value as well.

2.3 Structure and function of reference electrodes

The potential of the reference electrode is the reference point of the ion sensitive electrode the same way the sea level is the basis for altitude specifications. This potential develops in the limit area (see Fig. 7) between the electrolyte of the reference electrode and the test sample and can be measured by the meter using an inner reference electrode. The limit area is often stabilized by porous materials, the so-called junctions. There are different constructions guaranteeing sufficient contact with the sample, e.g. based on ceramic, fibers or glass frits.

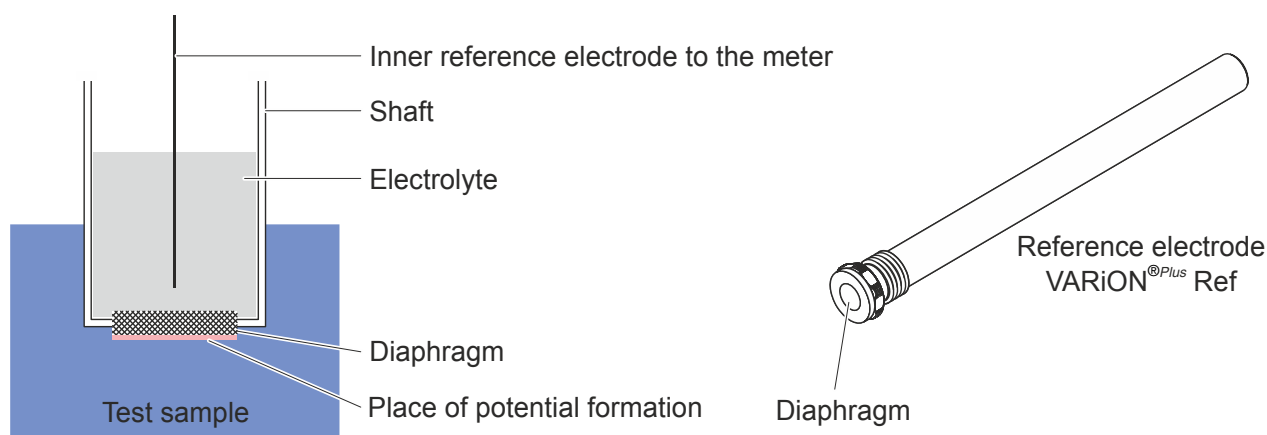


Fig. 7 Reference electrode (diagram on the left, VARiON^{®Plus} 700 IQ Ref model on the right))

During operation the potential of a reference electrode can slowly change due to lixiviation, aging or poisoning. This is also a basic reason for why one has to calibrate from time to time.

The junction is also responsible for the so-called diffusion voltages. They occur because the ion types present at this interface diffuse at different speeds. They cannot be completely eliminated. It is possible to keep them stable in a wide range by means of a suitable construction. The sample matrix also plays a role and has to be taken into account when calibrating (see chapter 5).

2.4 Temperature compensation

The measuring process of an ion sensitive electrode is subject to temperature effects. The slope S , for example, is considerably influenced by the temperature (see appendix, equation 2). WTW process systems therefore have an integrated temperature sensor. The thus determined temperature value is automatically included in the calculations while calibrating and measuring.

3 Application

3.1 ISE in online applications

Ion sensitive electrode have for a long time been used for laboratory analyses. Under these conditions the effect of different activities (see appendix) and diffusion voltages can be eliminated by adding ionic strength adapters or conditioning media.

If you want to use these techniques for online applications an automat has to be used. It can carry out the sample preparation and all measurement steps automatically. This, however, requires a considerably higher device-related complexity.

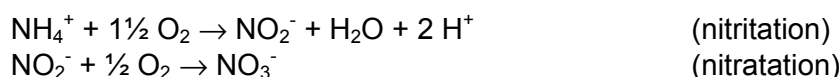
WTW has enhanced ISE technique and its handling so that it does not require such a high complexity now and can still be used over long periods. The VARiON^{®Plus} 700 IQ sensors provide reasonably priced and reliable measuring systems. As so-called in-situ systems they measure directly in test sample and the sample does not have to be pumped to an analysis location far away.

Nowadays their main application is the tracing of the cleaning process in waste water treatment plants. Thus it is possible to supplement D. O. measurement and to achieve a more effective waste water purification and energy saving in the field of nitrification / denitrification.

3.2 Principle of nitrification / denitrification

The first target of the biological cleaning steps in waste water treatment plants is to reduce the carbon load of the waste water. Nowadays, nitrogen compounds have to be reduced as well.

With the aid of the VARiON^{®Plus} 700 IQ sensors it is possible to trace and control the nitrification and denitrification processes happening in the basin. Nitrification runs in two steps. The first step is referred to as nitritation, where nitrosomonas bacteria oxidize ammonium to nitrite. In the second step (nitratation) nitrite reacts to nitrate with the involvement of nitrobacteria.



The following denitrification under lack of oxygen and in the presence of carbon as a reducing agent causes the formation of elemental nitrogen, which escapes into the atmosphere.



There are different possibilities of realizing nitrogen reduction in waste water treatment plants. Fig. 8 demonstrates the principle of intermittent nitrification / denitrification. The processes run one after the other in the same basin.

The standard carbon reduction takes place at the same time and independently of these processes.

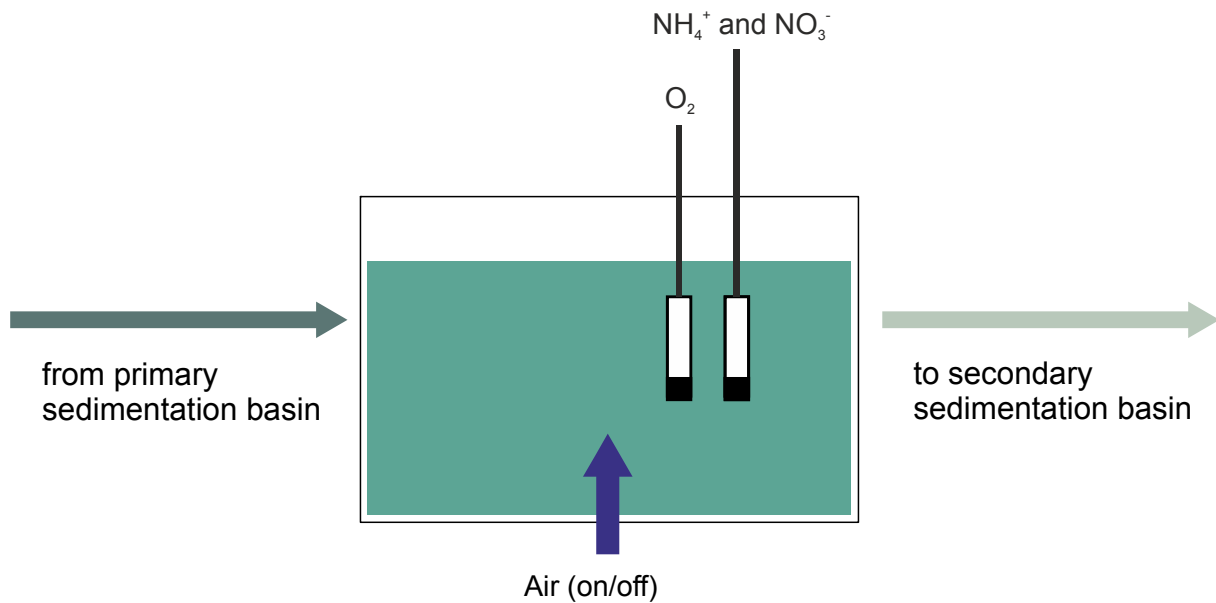


Fig. 8 Plant for intermittent operation in an activated sludge basin

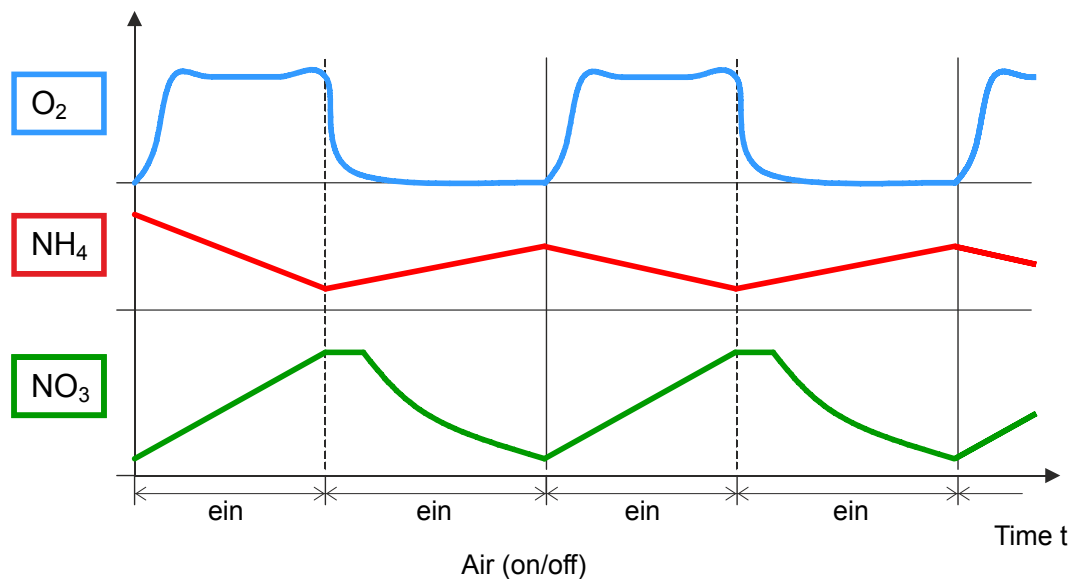


Fig. 9 Temporal course of nitrogen elimination with intermittent aeration.
Concentrations of D. O., ammonium and nitrate

As shown in Fig. 9, the D. O. content rises first due to aerating the activated sludge basin. Consequently, nitrification takes place. The ammonium concentration is reduced, the nitrate concentration is increased. The conditions are reversed in the denitrification phase. As soon as the nitrate has disappeared the process starts again.

Monitoring with the aid of ammonium and nitrate sensitive electrodes has proven to be most convenient. Thus the cleaning process can be optimized, for example by adjusting the switching points of the D. O. control via the ammonium value. The relevant control strategies always have to be realized in a way that the operational safety of the plant is guaranteed. The selection of the parameter best suited depends on the entire cleaning process and the respective measuring task.

Most frequently one value is used as the main parameter, the other for control purposes.

The nitrification and denitrification processes can also be carried out in separate basins. These processes run as preceding denitrification or successively added denitrification. Which procedure to select depends on the local conditions. The measurement and control parameters ammonium and nitrate are the same, however, with all procedures and can be monitored with the in-situ sensors.

4 ISE electrodes and IQ SENSOR NET

The ion sensitive electrodes (ISE) have to be mounted in an armature suitable for use in practice. This is enabled by the VARiON^{®Plus} 700 IQ, the AmmoLyt^{®Plus} 700 IQ and the NitraLyt^{®Plus} 700 IQ sensor.

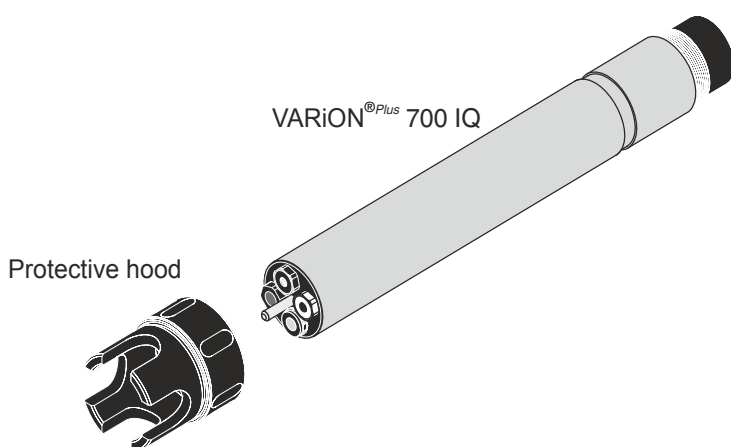


Fig. 10 VARiON^{®Plus} 700 IQ

The VARiON^{®Plus} 700 IQ is part of the sensor program provided by WTW within the IQ SENSOR NET. The VARiON^{®Plus} 700 IQ can determine the main parameters ammonium and nitrate using ion sensitive electrodes (ISE). One type of interfering ions (potassium or chloride) can be determined additionally and used for compensation purposes. A reference electrode serves as the common reference point for all ISE measurements.

The sensors AmmoLyt^{®Plus} 700 IQ for ammonium and NitraLyt^{®Plus} 700 IQ for nitrate operate on the same principle as the VARiON^{®Plus} 700 IQ. However, they only cover one of the two main parameters including the respective compensation. For better readability, only the VARiON^{®Plus} 700 IQ sensor is mentioned in this primer.

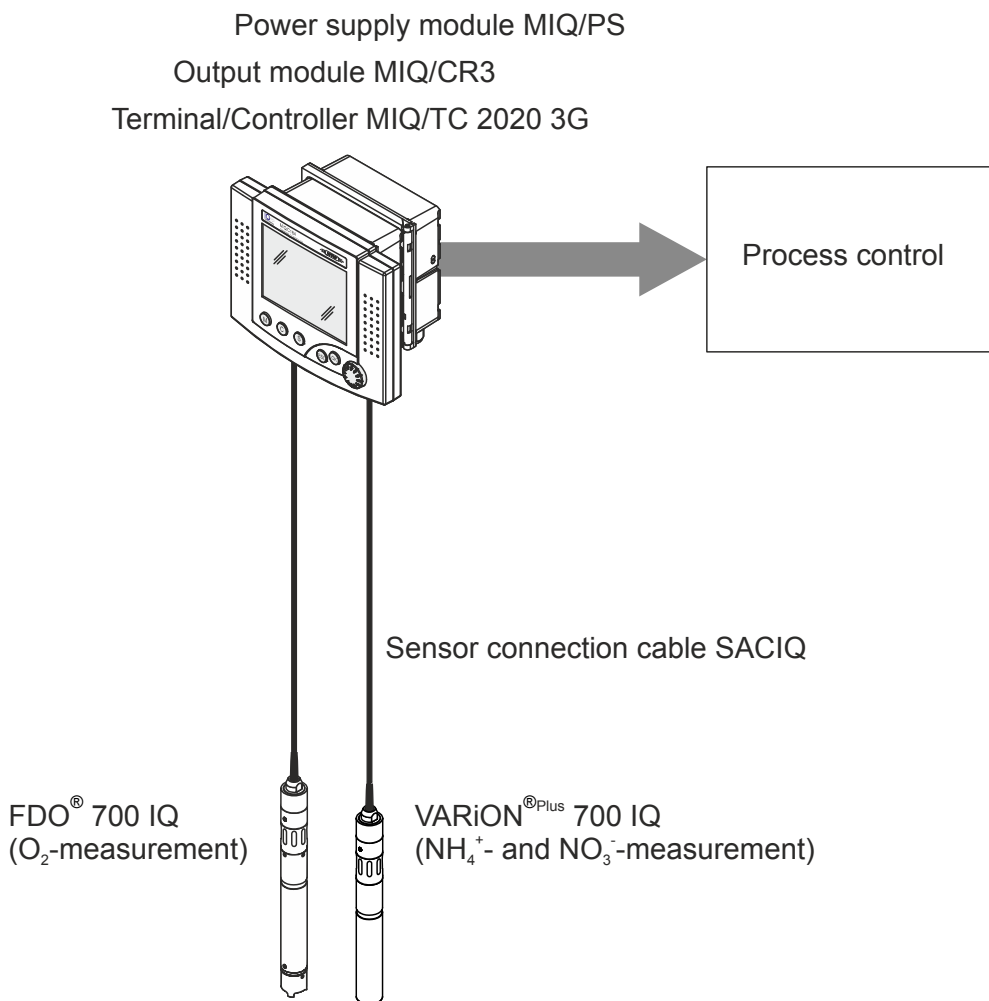


Fig. 11 Simple IQ SENSOR NET System for control in the aeration tank

The IQ SENSOR NET supplies the connected sensors with power. The sensor parameters can be centrally adjusted and the measured values displayed at a terminal even if the sensors are distributed over the entire plant. The VARiON^{®Plus} 700 IQ sensor with its ion sensitive electrodes is a unit that is integrated in the IQ SENSOR NET as an ammonium or nitrate sensor or as both. Depending on the tasks, the system can be supplemented by the VARiON^{®Plus} K or VARiON^{®Plus} Cl compensation electrodes.

5 Calibration and measurement

5.1 Purpose of calibration

Like any measuring system, ISE systems have to be calibrated from time to time. This procedure proves the operability of the system. It determines the sensor characteristics that might be changed by aging and that are used to calculate the concentration values from the raw measured values. The calibration data also provide information for the cleaning of the electrodes or finally, for their exchange.

To use the advantages of ISEs it makes sense to work direct-potentiometrically, i.e. to use the signals of the electrodes positioned in the measuring medium without manipulating the medium. To guarantee the measuring operation the following two calibration procedures are used:

- Two-point calibration in standards
- Single-point calibration using a reference value

5.2 Two-point calibration in standards

As can be seen in the equation (3), there is a linear relation between the logarithm of the concentration and the measured voltage. Two voltage concentration value pairs are sufficient to describe this relation. Thus it is sufficient to consecutively immerse the sensor in two different standard solutions to determine the values for S and U_{ion}^0 . These standards must be especially adapted for the conditions of ISE measurement. Standards used for other purposes cause erroneous calibrations when used here. Standards for the simultaneous calibration of all parameters are available for multiparameter systems like the VARiON^{®Plus} 700 IQ. The effects of interfering ions are taken into account.

When calibrating for the first time the value U_{ion}^0 is stored as the reference point. All following calibrations output the difference between the then determined U_{ion}^0 values and this point as the drift voltage. The determined slope standardized to 25 °C and the drift voltage indicate the condition of the measuring system. Contamination can cause a slow adjustment behavior and reduced slope values. If this is not improved by cleaning, the aging of the membrane can also be the cause, e. g. due to loss of the ionophore. If the limit value of the slope is undercut the ISE must be replaced. The developing of the drift voltages is mainly caused by the aging of the reference electrode. It has to be replaced if the drift voltage is outside the valid range.

For a successful calibration it is important to follow the given procedures. These are the conditioning, discarding the conditioning solution, temperature adaptation and stabilization of measured values in standard solutions. The correct order of the standard solutions accelerates the procedure.

Two-point calibration is carried out in standard solutions with defined concentrations of the ions to be measured. It cannot reproduce the conditions of the matrix, d. h. the composition of the sample and its effect on the measured ions activity and diffusion voltages in the actual test sample, as these are different for each application and can change with time. A matrix adjustment is possible with the single-point calibration using a reference value.

5.3 Single-point calibration using a reference value

Single-point calibration using a reference value is for matrix adjustment. It is carried out after each two-point calibration or can be used individually from time to time. During this calibration procedure the sensor is not in a standard but in the test sample. At the same time, the current measured value for the relevant parameter is determined with a second, independent measurement procedure. A photometrical test is normally used for this. The adjustment for the sample matrix is done for U_{ion}^0 while maintaining the slope from the two-point calibration. The advantage is that all matrix effects are included while calibrating. If interfering ions are present that should be manually compensated for, their concentration also have to be determined and entered on the measuring system.

5.4 Recommendations for calibration in practice

A two-point calibration is normally carried out for control purposes at bigger intervals, e.g. once a month. A single-point calibration should also be carried out in between, e.g. if you assume that the sample composition has changed considerably. It can be done quite easily as the sensor is left in the basin for this.

When sampling for the comparison value make sure the measurement is done immediately or the sample is stabilized. The sample contains microorganisms and, therefore, the ammonium and nitrate values change very quickly.

6 Measuring

6.1 Assembly

The position of the sensors should ensure the data later measured is representative for the required task. The installation depends on the respective conditions and has to be adjusted to the local requirements. Freely oscillating installation on a chain is always better than fixed installation. Thus the sensor can move in the basin and clean itself to a certain degree. When the cleaning system is used a compressed air supply has to be provided.



Fig. 12 VARiON^{®Plus} 700 IQ in the activated sludge basin

6.2 Citation form

The measurement data can be displayed and documented in different ways. The ammonium concentration can either be stated as the NH_4^+ ammonium value or as the $\text{NH}_4\text{-N}$ ammonium nitrogen value. It is important to be aware of the citation form that is used. With the parameter ammonium, a mix-up will not clearly stand out due to the similar molecular weight. With nitrate, the difference is, however, considerable. A nitrate NO_3^- concentration of 10 mg/L corresponds to a nitrate nitrogen $\text{NO}_3\text{-N}$ value of only 2.3 mg/L.

6.3 Impact of the pH value on ammonium determination

The pH value of the test sample affects the chemical balance between NH_4^+ and NH_3 .



From a pH value of 7, the NH_4^+ part of the test sample decreases and the NH_3 part increases with a rising pH value. NH_3 is not detected by the ammonium electrode.

NH_4^+ part
in the solution in %

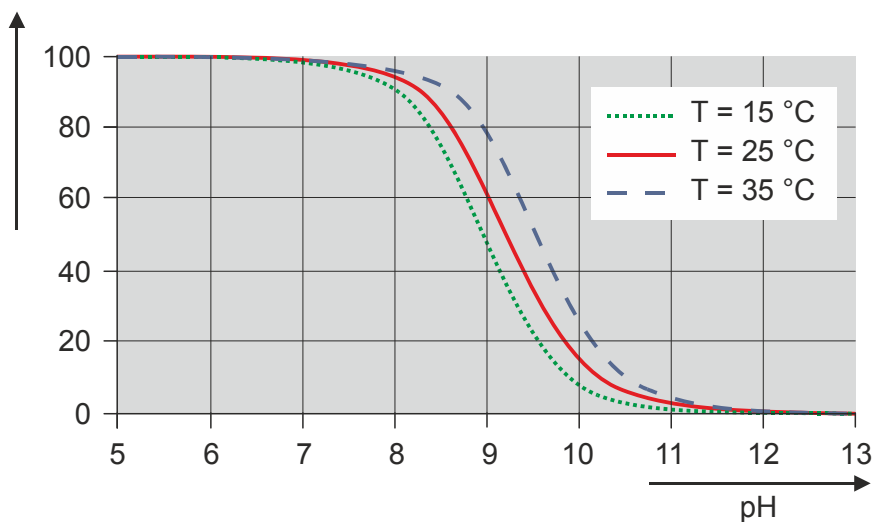


Fig. 13 Ammonia balance

For measurements in test samples with a pH value > 7 , a compensation for the influence of the pH value may be necessary. The compensation can for example be carried out by the higher process control with an additional pH measurement.

Slightly higher pH values can be taken into account in the calibration procedure if necessary.

6.4 Usage in practice

The quick availability of the data measured directly in the medium using an ISE sensor can be seen in Fig. 14, using ammonium measurement as an example.

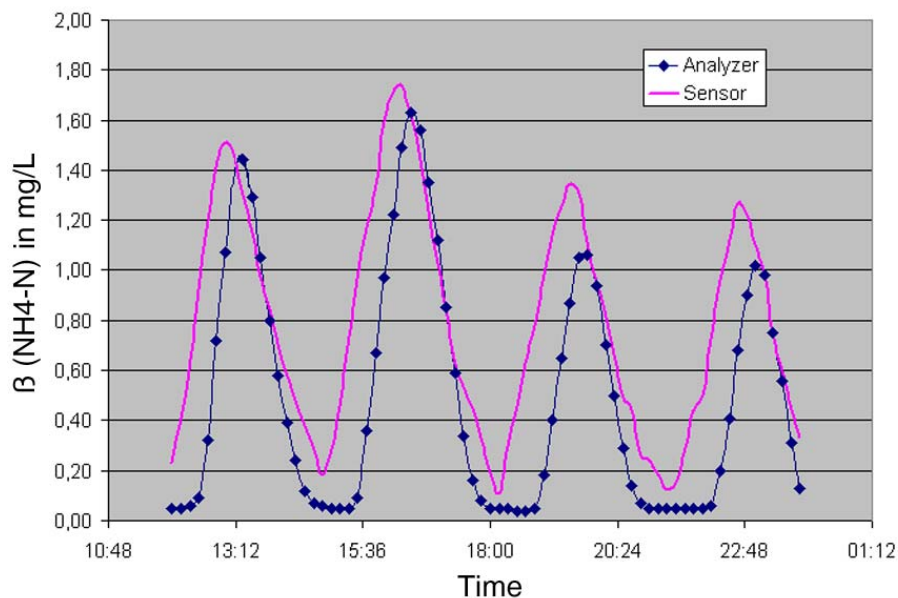


Fig. 14 Ammonium measurement with ISE (continuous line) and analyzer (dotted line)

The in-situ system with ISE clearly reacts quicker to changes of the measuring medium than an analyzer with sample preparation. The time delay for this example is approx. 15 minutes.

How stable such system run is shown in Fig. 15, where a nitrate measurement is compared to an analyzer.

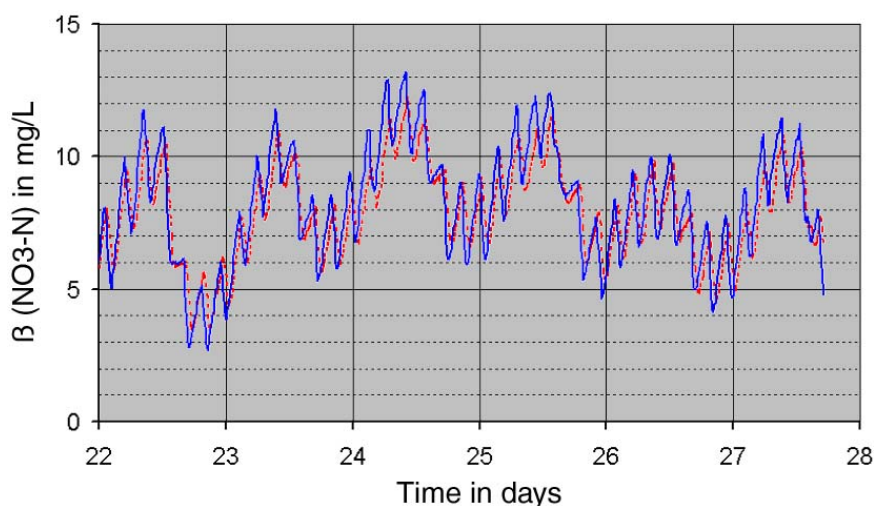


Fig. 15 ISE online data (continuous line) and analyzer (dotted line) without recalibration of the nitrate sensor

The ISE system was adjusted at the beginning of the measurement (day 0). The data of both systems well agree with each other without the necessity to recalibrate the electrodes.

7 Cleaning and maintenance

Usage in sludge-containing media can cause contamination. To keep the sensors permanently operable is recommended to use a cleaning device. Like other sensors of the WTW program, the VARiON^{®Plus} 700 IQ can be equipped with the CH cleaning head, which enables interval-controlled compressed air cleaning in the medium.



*Fig. 16 Left: CH cleaning head to screw on the VARiON^{®Plus} 700 IQ
Right: Successfully keeping free the measurement electrodes during operation*

This cleaning method is normally sufficient for synthetic material electrodes. With solid body electrodes, the membrane sometimes has to be polished with the polishing strip provided.

When being stored the electrodes have to be kept moist in the solutions intended for this.

8 Operating costs

Due to feedback control with ISE measuring technique, the blowing of air into the activated sludge basin is optimized. The blowers only operate as long as is necessary for the required degradation of the carbon and nitrogen load. Thus the energy costs are reduced. The investment in ISE measuring technique will quickly pay for itself (see also WTW application report, AMMONIUM - CONTROL OF N-ELIMINATION VIA NH₄-N CONSIDERING THE BIBERTAL-HEGAU WASTE WATER TREATMENT PLANT AS AN EXAMPLE).

9 Summary

The VARiON^{®Plus} 700 IQ measures the two most important parameters in the activated sludge basin: ammonium and nitrate. Together with a D. O. measurement, all important parameters for controlling the cleaning process are thus available. The advantages of the VARiON^{®Plus} 700 IQ are the short response times and the availability of real time data provided by in-situ measurement. Transporting and preparing the sample is not required. All sensors can be easily integrated into the control process of the plant via the IQ SENSOR NET.

The VARiON^{®Plus} 700 IQ automatically compensates for the temperature influence and is not influenced by coloration or turbidity due to the measuring principle. Matrix-related impacts on the measurement are eliminated by means of a single-point calibration. The interfering ions potassium or chloride are automatically compensated for by the VARiON^{®Plus} 700 IQ.

The VARiON^{®Plus} 700 IQ automatically recognizes the different electrode types during commissioning. When the corresponding electrodes are mounted the interfering ions compensation is automatically activated. The electrodes have a long working life and can easily be replaced if necessary.

Generally, using the VARiON^{®Plus} 700 IQ in the activated sludge basin optimizes the process and leads to lower costs of investment and operation (more efficient use of energy). At the same time the municipal waste water charges are reduced due to the improved water quality in the outlet of the waste water treatment plant.

10 Appendix

Nernst equation

For ISE, the Nernst equation describes the relation between the measured ions contents and the displayed voltage

$$U_{ion} = U_{ion}^0 \pm S \cdot \log(a_{ion}) \quad (A1)$$

where U_{ion} potential of the electrode
 U_{ion}^0 standard potential of the electrode at a reference point
 \pm arithmetic sign (+ for positively charged ions and – for negatively charged ions)
 S slope of the electrode (depending on the valency of the ions)
 \log logarithm of the basis 10
 a_{ion} activity of the relevant ion type

Due to their positive charge, cations produce a slope of the line, anions a negative slope.

The slope can be specified more exactly with

$$S = \frac{RT}{zF} \quad (A2)$$

where R gas constant ($8.314 \text{ J} \cdot \text{K}^{-1} \cdot \text{mol}^{-1}$)
 T temperature in Kelvin
 z valency of the ions
 F Faraday constant ($96485 \text{ C} \cdot \text{mol}^{-1}$)

Here the impact of the temperature becomes clear. The theoretical value of the slope (so-called Nernst slope), is - according to amount - 59.16 mV for 25 °C for simply charged ions, i.e. with a change of the activity by the factor 10 the voltage changes by this amount.

To take the interfering ions into account, the argument in brackets of the logarithm from the equation (A1) is extended by the dimensionless selectivity coefficient $K_{meas, interf}$ and the interfering ions activity a_{interf} .

$$U_{ion} = U_{ion}^0 \pm S \cdot \log(a_{ion} + K_{ion, st\ddot{o}r} \cdot a_{st\ddot{o}r}) \quad (A3)$$

The activity of an ion results from its contents multiplied by the activity coefficient valid for the relevant solution. The activity coefficient depends on the total ion content of a solution. It equals one for very diluted solutions. With higher concentrations it is smaller. The interrelationships in real solutions can be very complex and activity values are merely available. Therefore, one has to use suitable procedures as described in this primer for calibrating and measuring procedures.

Xylem |'zīləm|

- 1) The tissue in plants that brings water upward from the roots;
- 2) a leading global water technology company.

We're a global team unified in a common purpose: creating advanced technology solutions to the world's water challenges. Developing new technologies that will improve the way water is used, conserved, and re-used in the future is central to our work. Our products and services move, treat, analyze, monitor and return water to the environment, in public utility, industrial, residential and commercial building services settings. Xylem also provides a leading portfolio of smart metering, network technologies and advanced analytics solutions for water, electric and gas utilities. In more than 150 countries, we have strong, long-standing relationships with customers who know us for our powerful combination of leading product brands and applications expertise with a strong focus on developing comprehensive, sustainable solutions.

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Xylem Analytics Germany
Sales GmbH & Co. KG
WTW
Am Achalaich 11
82362 Weilheim
Germany

Tel.: +49 881 183-325
Fax: +49 881 183-414
E-Mail wtw.rma@xylem.com
Internet: www.xylemanalytics.com



Xylem Analytics Germany GmbH
Am Achalaich 11
82362 Weilheim
Germany