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Primer: Ion selective measurement in online analysis

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Contents

Fundamentals of the ion selective measuring technique	4
1.1 The principle	4
1.2 The Nernst equation	5
2 Electrodes.....	6
2.1 Structure and function of ion sensitive electrodes	6
2.2 Influence of interfering ions and selectivity coefficient.....	8
2.3 Structure and function of reference electrodes.....	10
2.4 Temperature compensation.....	10
3 Application	11
3.1 ISE in online applications.....	11
3.2 Principle of nitrification / denitrification.....	11
4 VARiON system and IQ SENSOR NET.....	13
5 Adjusting, checking, calibrating	15
5.1 Freedom from calibration.....	15
5.2 Matrix adjustment	15
5.3 Procedure for special cases	16
5.3.1 Check.....	16
5.3.2 Calibration.....	16
5.4 Drift voltage, electrode zeroing, calibration history	16
6 Measuring.....	17
6.1 Assembly	17
6.2 Citation form	17
6.3 Influence of the pH value on ammonium determination	18
6.4 Usage in practice	18
7 Cleaning and maintenance.....	20
8 Operating costs	20
9 Summary	21
10 Appendix.....	22

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 about individual substances using 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 YSI developments demonstrate that this measurement principle is also suitable for 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).

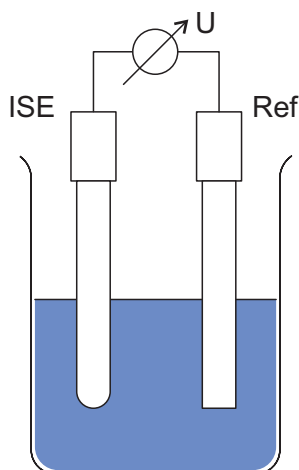


Fig 1 Schematic structure of an ion sensitive measuring system with ISE (ion sensitive electrode), Ref (reference electrode) and voltmeter

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, a plus is for positively charged cations, e.g. K^+ , a minus for anions such as Cl^- .

The activity a is the active concentration of an ion type that takes into account the influence 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

$$U_{ion} = U_{ion}^0 \pm S \cdot \log(\beta_{ion}) \quad (3)$$

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.

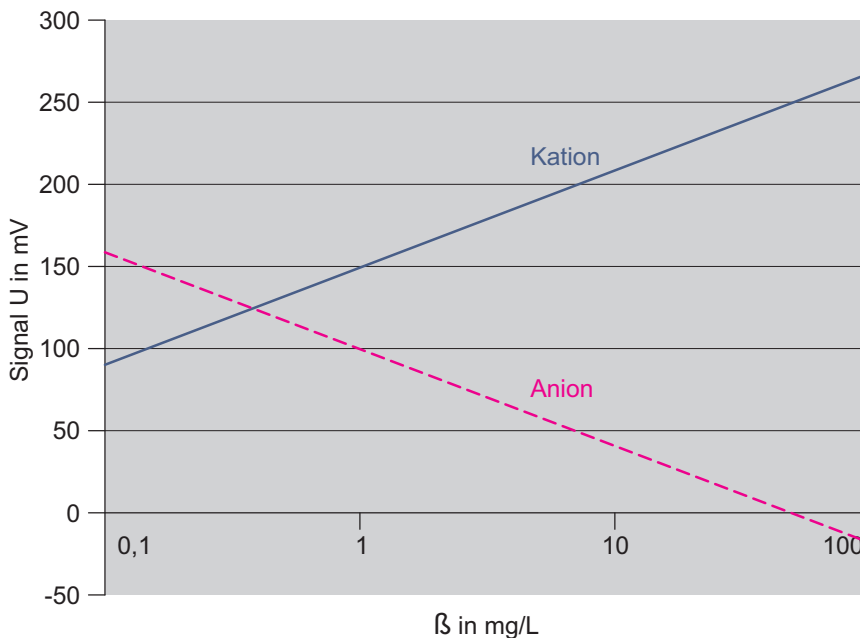


Fig 2 Theoretical calibration lines for anions and cations (according to Nernst equation)

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.

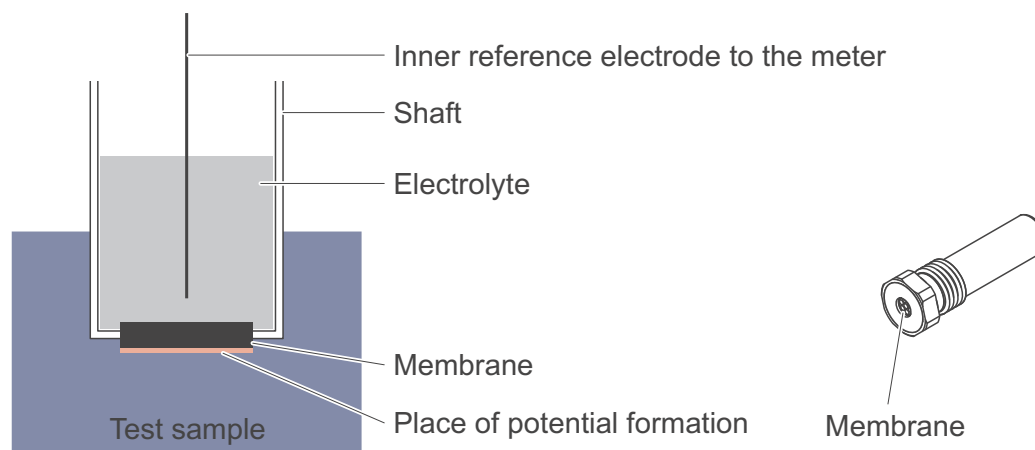


Fig 3 Structure of an ion sensitive electrode (diagram on the left, sample model on the right)

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

Glass membranes 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).

Other ions, e.g. Na^+ can also be determined with the corresponding glass types and special procedures.

Solid body membranes 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.

Synthetic material membranes 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.

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

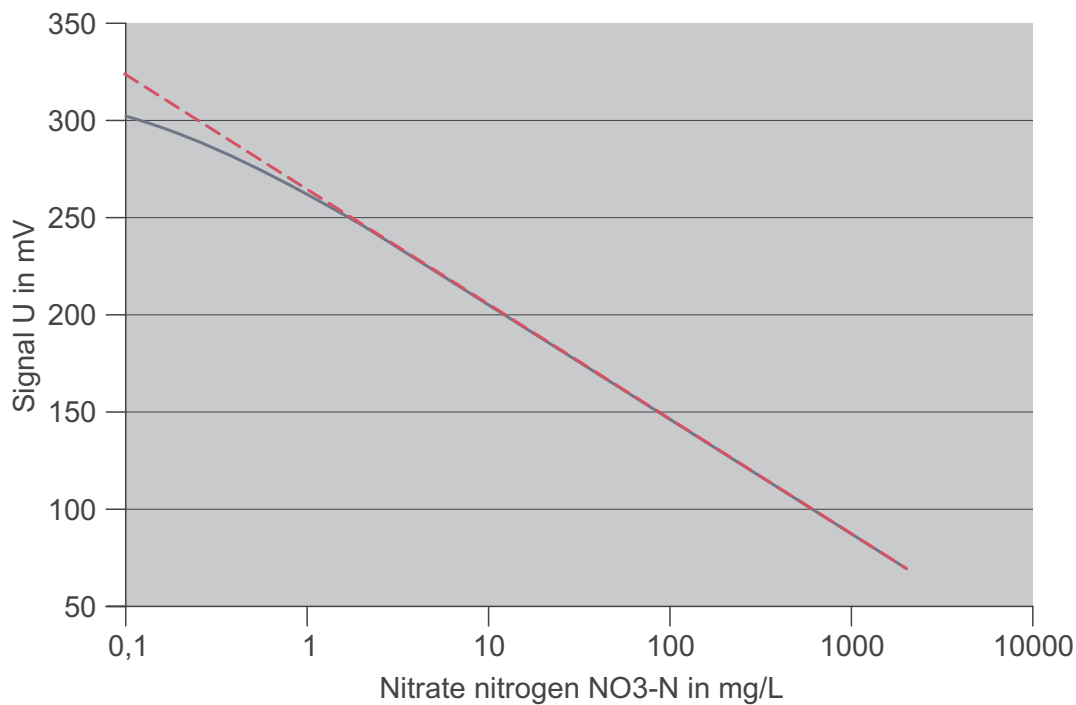


Fig 4 Real characteristic curve of a nitrate ISE (solid line) and purely theoretical Nernst function (dashed)

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 YSI measuring systems this effect is taken into account automatically. If the concentrations are even 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 Influence of interfering ions and selectivity coefficient

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 chemically acts similar. 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 $K_{\text{meas,interf}} = 0.2$ states that the interfering ion produces the same voltage as the concentration of the measured ion multiplied by 0.2. If $K_{\text{meas,interf}} = 0.01$, 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 system, 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. The VARiON system provides this option. Fig 5 shows the interrelationship of the 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.

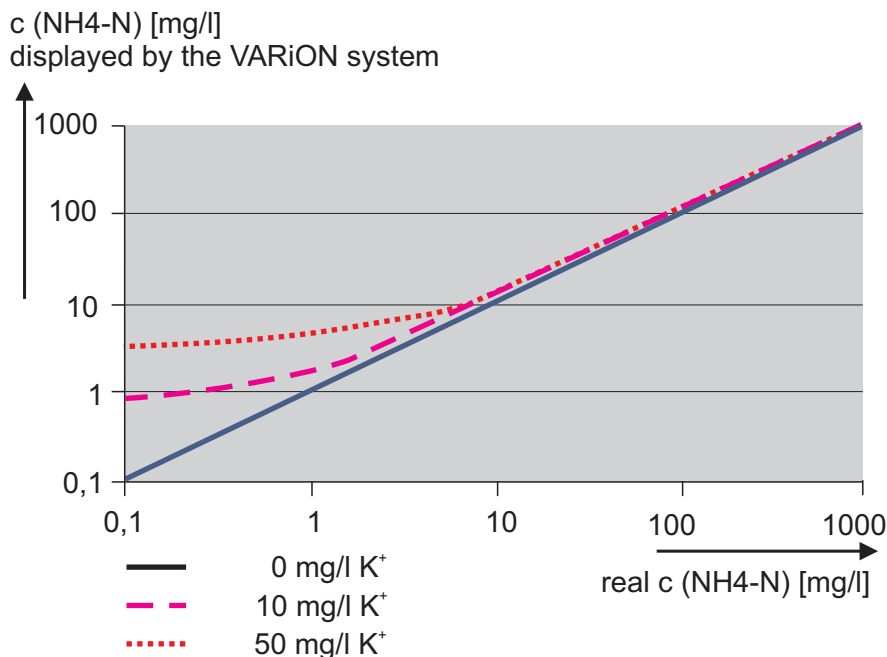


Fig 5 Influence of potassium on the ammonium value

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 system:

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

The relevant curves are shown in Fig 6.

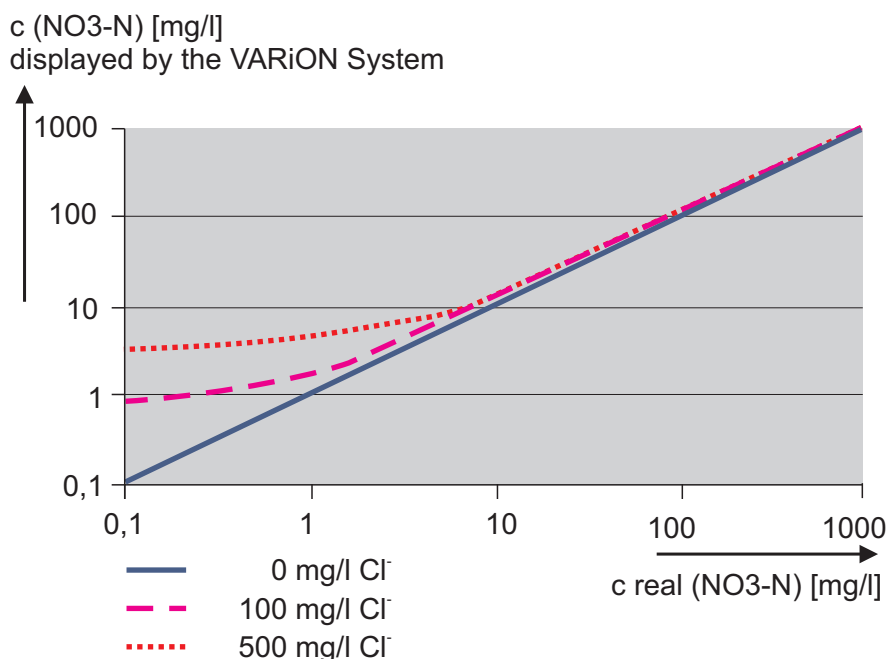


Fig 6 Influence of chloride on the measured nitrate value

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 due to 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 additionally.

2.3 Structure and function of reference electrodes

The potential of the reference electrode is the reference point of the ion sensitive combination 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.

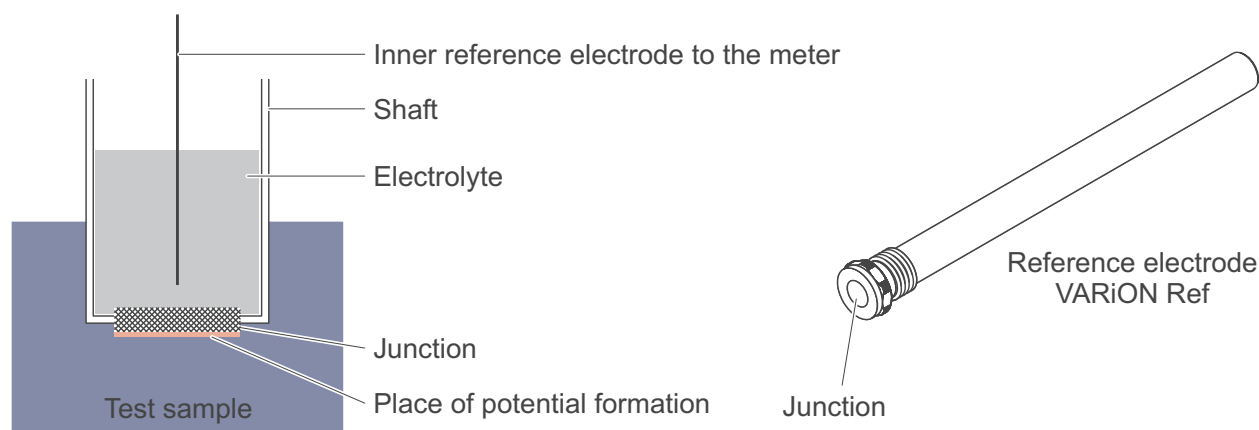


Fig 7 Reference electrode (diagram on the left, VARION Ref model on the right)

During operation the potential of a reference electrode can slowly change due to lixiviation, aging or poisoning. This can be counteracted by calibrating. The VARION Ref was designed so that these effects are not relevant in its main applications during its operational lifetime.

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. The design of the VARION Ref stabilizes this influence over a wide range. The effect of different sample compositions, the so-called matrix effect, is counteracted by the matrix adjustment (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 A2). Therefore, YSI online systems 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 electrodes 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 complexity of devices.

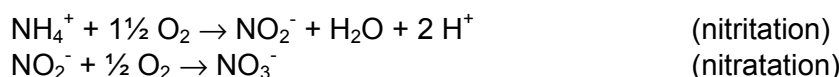
YSI has enhanced ISE technique and its handling so that it does not require such a high complexity now and can still be used over long periods. The VARiON system provide reasonably priced and reliable measuring systems. As so-called in-situ systems they measure directly in the test sample and the sample 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, which has been used for a long time, 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 system it is possible to trace and control the nitrification and denitrification processes taking place 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.

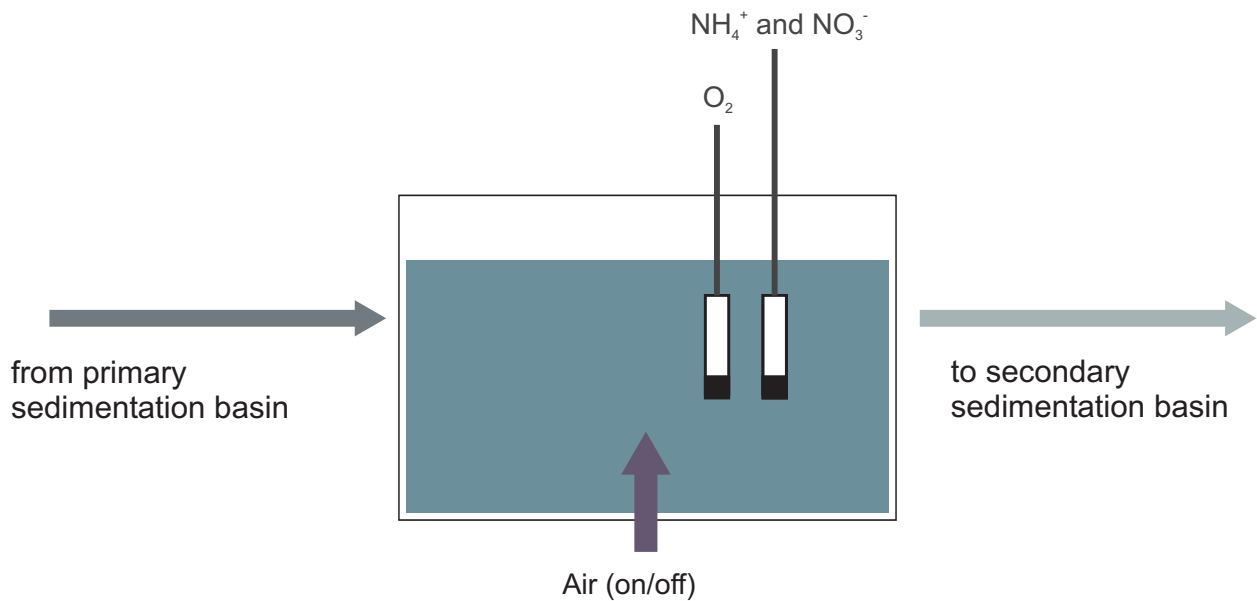


Fig 8 Plant for intermittent operation in an activated sludge basin

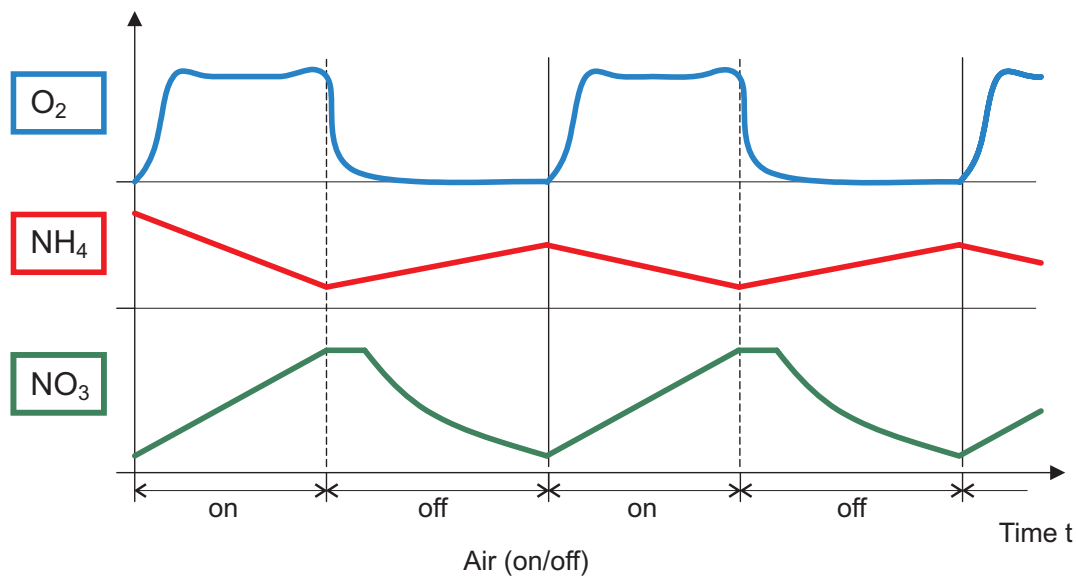


Fig 9 Temporal course of nitrogen elimination with intermittent aeration. Concentrations of D. O., ammonium and nitrate

As shown in Fig 9, the D. O. content rises first due to aeration of 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 guarantees the operational safety of the plant. 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 with all procedures, however, and can be monitored with the in-situ sensors.

4 VARiON system and IQ SENSOR NET

The ion sensitive electrodes have to be mounted in an armature suitable for use in practice. This is enabled by the VARiON system.

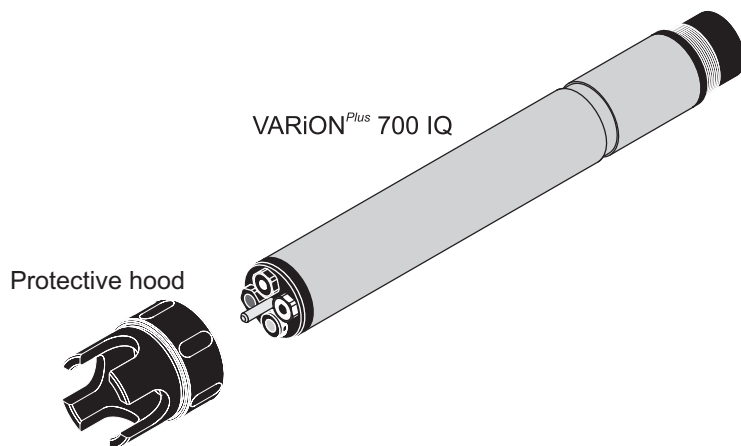


Fig 10 VARiON^{Plus} 700 IQ

The VARiON system is part of the sensor program provided by YSI within the IQ SENSOR NET. The VARiON system 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.

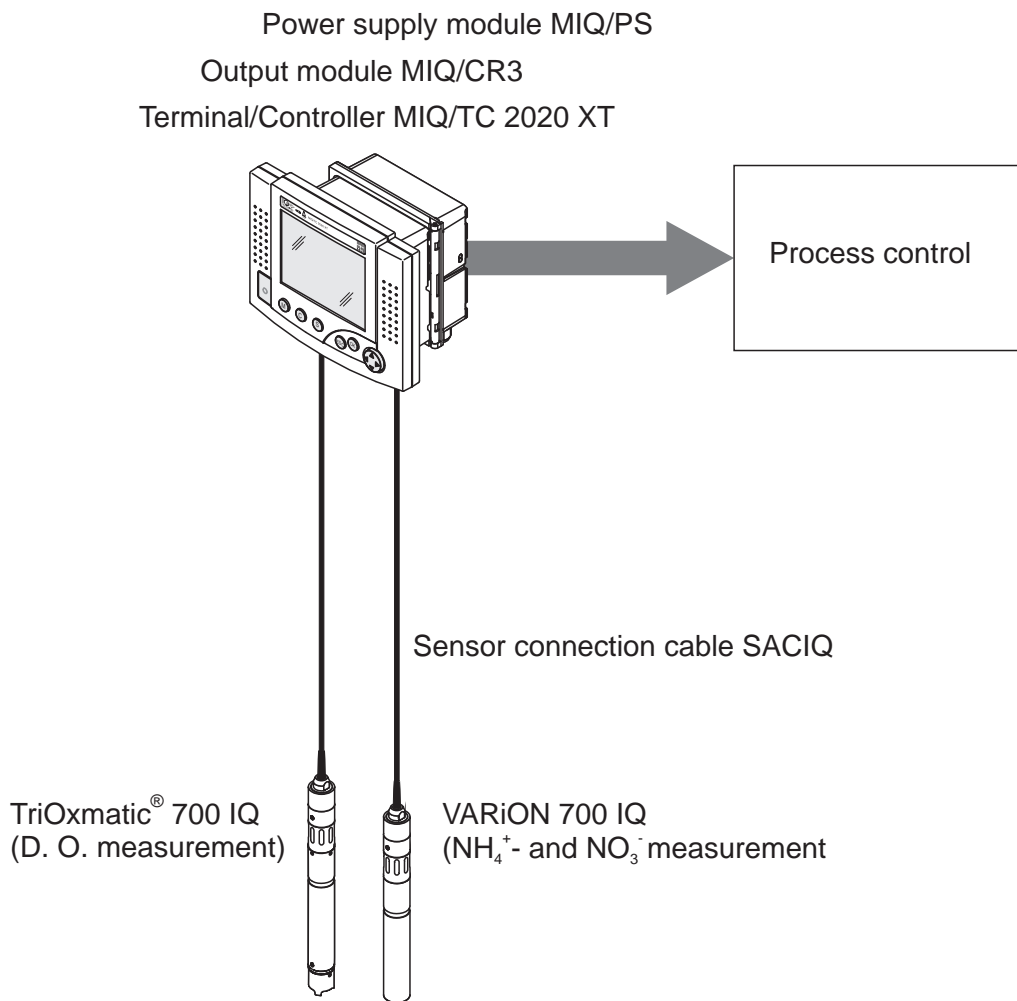


Fig 11 Simple IQ SENSOR NET system for control in the activated sludge basin

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 VARIION system with its ion sensitive electrodes is a unit that is integrated in the SENSOR NET as an ammonium or nitrate sensor or as both. Depending on the tasks, the system can be supplemented by the VARIION^{Plus} K or VARIION^{Plus} CI compensation electrodes.

5 Adjusting, checking, calibrating

5.1 Freedom from calibration

Normally, the influences on the characteristic data of an electrode that exist with potentiometric measuring procedures are counteracted by calibration. Calibration redetermines the characteristic variables, and the measuring system is adjusted to the changed conditions.

With the electrodes for the VARIION system, YSI has reached a high technical state. The ion sensitive electrode and reference electrodes are stable for their operational lifetime regarding the typical applications and thus practically calibration free. This is demonstrated by the preservation of the slope of the ISE and the reference potential of the reference electrode.

Even stable systems have to be checked once in a while. The check proves the operability of the system. Clues for cleaning or eventually exchanging the electrodes result from the check data.

To use the advantages of ISEs it makes sense to work direct-potentiometrically, i.e. to use the signals of the sensor positioned in the measuring medium ("in-situ") without changing the medium. The metrological stability of the electrode enables a simplified process according to the procedures for guaranteeing the measuring operation shown below:

- Matrix adjustment
- Check
- Calibration

The software guides through the processes and supports the user by an online help.

5.2 Matrix adjustment

The matrix adjustment is the usual procedure for operating the sensors. As every application has its own features based on the composition of the test sample, the matrix adjustment has to be carried out after commissioning the measuring system. Even later the system is from time to time adjusted to major changes of the sample composition by means of this procedure.

The procedure is very simple because the sensor remains in the test sample. It stores the relevant data while the current measured value for the relevant parameters is being determined by a second, independent measuring procedure at the same time. A photometrical test is normally used for this. Consequently, the adjustment with the sample matrix is carried out for U_{ion}^0 . The advantage is that all matrix effects are included while adjusting.

If interfering ions are present that should be manually compensated for, their concentrations also have to be determined and entered on the measuring system.

For mere test purposes, the procedure can be reduced so that just a comparison value is determined with a second procedure. A complete matrix adjustment is only carried out if the displayed measured value deviates from the comparison value by more than the specified tolerance.

5.3 Procedure for special cases

5.3.1 Check

The check is advisable if measured values seem to be implausible or within the scope of regular testing. It is a simple procedure to provide evidence of the operability of the electrodes used. The procedure takes place in two standard solutions of different concentrations and results in "+", i.e. the electrode is OK, or "-", i.e. the electrode is possibly not OK. It is a quick test with limited accuracy. The results serve solely to evaluate the electrode and do not change any characteristics or sensor settings.

5.3.2 Calibration

A calibration meaning the exact measuring of two standard solutions with different concentrations is not required in normal operation. It can make sense if the measured values do not seem to be correct after a matrix adjustment and/or check, if the exact electrode slope should be determined or if the sample material is especially critical.

To perform the calibration it is sufficient to successively immerse the sensor in two different standard solutions. Equation (3) shows that there is a linear relationship between the logarithm of the concentration and the measured voltage, and that with this procedure two voltage concentration value pairs are obtained that serve to determine S and U_{ion}^0 .

The used 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 system. The effects of interfering ions are taken into account.

For successful calibration it is important to follow the given procedures. These are the conditioning, discarding of the conditioning solution, temperature adaptation and stabilization of measured values in standard solutions. The solutions have to be stirred at least temporarily. If a corresponding interface or separate measuring system is available, calibration can also be carried out in the lab.

Calibration is carried out in standard solutions with defined concentrations of the ions to be measured. It cannot reproduce the conditions of the matrix, i. e. 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. After carrying out calibration, it is always required to perform the matrix adjustment additionally.

5.4 Drift voltage, electrode zeroing, calibration history

The "drift voltage" serves to evaluate the long term behavior of an electrode in its application. To start the evaluation period, the user has to set the "drift voltage" to zero using the "electrode zeroing" function. This is usually done during the initial commissioning or when inserting a new or different electrode. The matrix adjustment and calibration procedures provide this option automatically. The temporal change of the "drift voltage" can be viewed in the "calibration history" of the relevant electrode. The "drift voltage" indicates changes of the matrix or the electrode characteristics.

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 Influence 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.

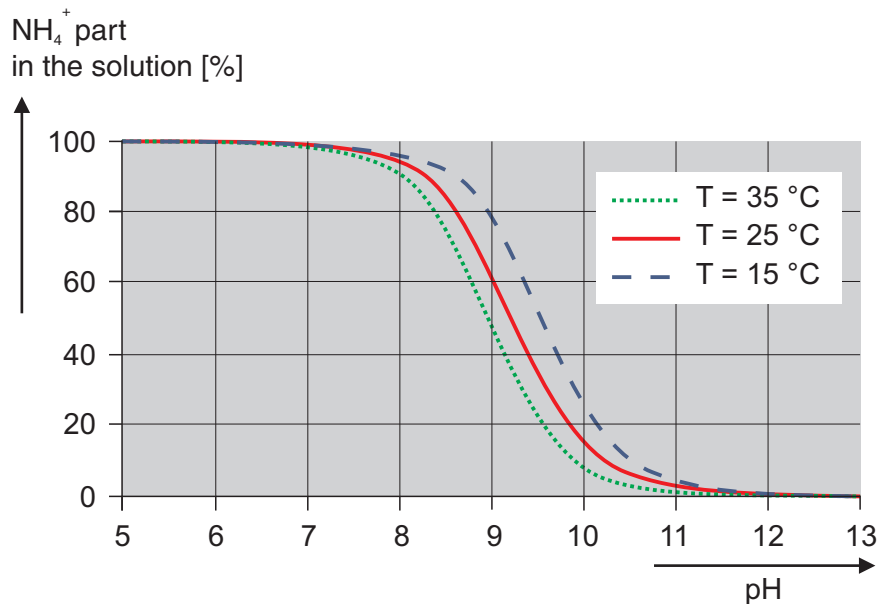


Fig 13 Ammonia balance

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.

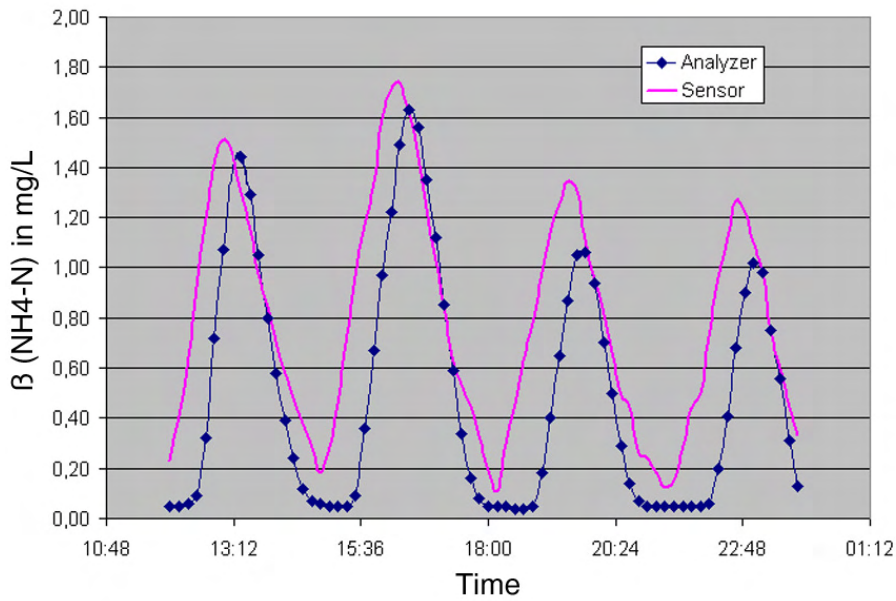


Fig 14 Ammonium measurement with ISE (continuous line) and analyzer (dotted line).

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 a system runs is shown in Fig 15, where a nitrate measurement is compared to an analyzer.

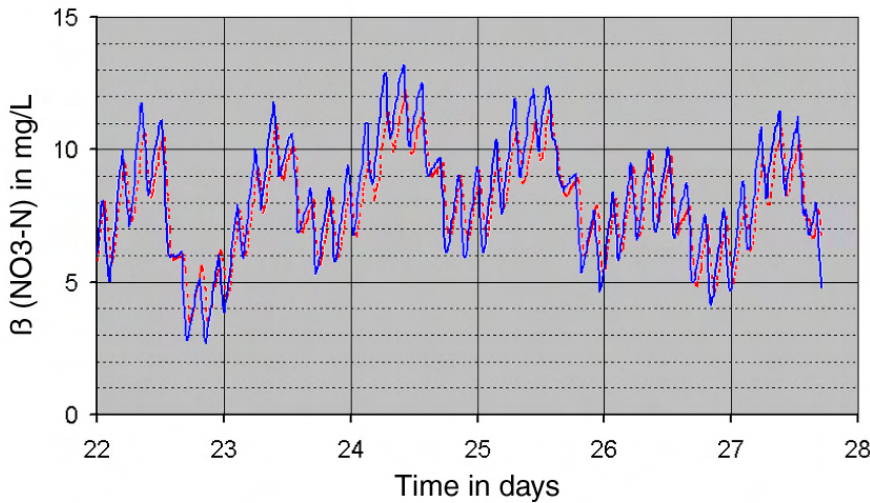


Fig 15 ISE online data (continuous line) and analyzer (dotted line) without recalibration of the nitrate sensor

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 it is recommended to use a cleaning device. Like other sensors of the YSI program, the VARiON system 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 be screwed on the VARiON system
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.

All maintenance activities can be conveniently carried out with the maintenance case, VARiON Case, which is available individually.

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.

9 Summary

The VARiON system measures the two most important parameters for nitrogen removal in the activated sludge basin: ammonium and nitrate. Together with D. O. measurement, all important parameters for controlling the cleaning process are thus available. The advantages of the VARiON system 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 system automatically compensates for the temperature influence and is not influenced by coloration or turbidity due to the measuring principle. Matrix-related influences on the measurement are eliminated by means of the matrix adjustment. The interfering potassium or chloride ions are recorded and dynamically compensated for by the VARiON system.

The VARiON system recognizes the different electrode types installed during commissioning. The installation of a compensation electrode activates the automatic interfering ions compensation. The electrodes are calibration free, have a long working life and can easily be exchanged if necessary.

Altogether, using the VARiON system in the activated sludge basin optimizes the process and leads to lower costs of investment and operation for the measuring system. The aeration control can be performed load dependent. This considerably reduces the energy costs. At the same time, the water quality in the waste water outlet is improved. This can lead to clearly reduced waste water charges.

10 Appendix

Nernst equation

For ISE, the Nernst equation describes the relation between the measured ions concentration 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 positive 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 influence 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 $K_{ion,interf}$ and the interfering ions activity a_{interf} .

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

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 solutions that are very much diluted. With higher concentrations it is smaller. The interrelationships in real solutions can be very complex and activity values are rarely available. Therefore, one has to use suitable procedures as described in this primer for calibrating and measuring procedures.



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