pH probe type 03/04 ring pH probe



Operating Instructions

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1 Preface

These operating instructions are designed to familiarize users with the equipment of the pH probe and its use.

The operating instructions should be accessible to the operating and maintenance personnel in order to ensure that all necessary information is available for any assembly and maintenance work.

By knowing these operating instructions, you can avoid damage to the measuring equipment and ensure trouble-free operation.

The information contained in these operating instructions correspond to the state of the art at the time it is printed and is provided to the best of our knowledge.

We reserve the right to include any improvements, amendments and new developments in the operating instructions without prior notice. The actual design of products may differ from the information provided in the catalog if warranted by technical modifications resulting from product improvements. The proposal submitted by Pfaudler for a concrete application will be binding in this case.

The latest edition will always supersede all previous ones.

The present operating instructions are made available to our customers and interested parties free of charge. Reprints and copies as well as transformation into electronic forms, in whole or in part, shall require our written approval.

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2 General instructions

2.1 Operating range

The glassteel pH probes are used to measure the pH value of a solution. The pH value is a measure of the strength of an alkali or acid solution. The pH probes are installed directly in the reactor or the pipeline. For the resistance of the probes, please refer to our publications no. 206 and 641.

Never operate this measuring device outside its permissible operating conditions

2.2 Warnings contained in the operating instructions

In the operating instructions, the danger symbol is used to draw your attention to especially important safety instructions. Compliance with these instructions is mandatory, because adherence to the instructions can avoid severe damage to people and/or equipment.

2.3 Notes on deliveries

The respective scope of delivery is specified on the shipping documents attached to the shipment and corresponds to the valid purchase agreement.

Check that the items delivered for complete and intact. If possible, keep the packing material for re-use for possible return shipments.

2.4 Transport and storage

The probes should only be transported and stored in their closed original packaging, if possible. Where this is not possible, the probes must be protected against shock and impacts. Use the eye bolts provided for suspending the probes (for pipe diameter of 83 mm or more). In order to guarantee an as-new condition of the probe, maintain the following storage conditions: dry and dust-free as well as steady temperature and ventilation. The probes do not need any preservatives, they are resistant to normal environmental influences.

Type 04 ph probes must not be rested on the probe tip!

In the event of long service interruptions, the measuring probe may be stored dry or remain inside the reactor. If the probe is to be stored for prolonged periods of time, the probe's electrolyte line must be rinsed with distilled water.

The electrolyte line of the pH probe must be additionally blown through if there is danger of frost.

Dry storage basically does not affect the function of the probe. Its characteristics are maintained in any case. However, the zero point and slope may be subject to slight change which may result in a measuring error of 0.2 pH max. This error can be corrected by reconditioning the probe (refer to Sect. 10.2). During operation of the probe, this error will also disappear after 2-3 hours.

If probes are immersed in liquid (product), the pH transmitter or the isolation amplifier must not be disconnected from mains for more than 1-2 hours. Otherwise, polarization at the measuring electrode may result in a zero drift which may eventually be outside the setting range of the pH transmitter. Where longer disconnection is necessary, it is important to disconnect the measuring circuit at the probe either by removing the plug from the reference electrode or at the probe's terminal box.

2.5 Warranty notes

Any warranty claims shall not be extended or limited by the information contained in the present operating instructions.

For the exact warranty conditions, please refer to the Terms of Sale of Pfaudler Werke GmbH as amended.

2.6 Notes on return deliveries

Before sending probes to the manufacturer or third parties for repair or other purposes, all parts must be cleaned and decontaminated.

To protect our staff and for insurance reasons, your return shipment must be accompanied by a clearance certificate on which you confirm that the probe was properly cleaned and decontaminated. You may obtain a form sheet for this purpose from us on request.

3 Safety

For detailed safety instructions and information concerning explosion protection, please refer to the end of the operating instructions.

3.1 Proper use

Any use of the probe for purposes other than described in the present operating instructions will adversely affect the safety and functioning of the measuring device and is therefore not allowed.

It is important to note the safety instructions applicable to the electrical systems and equipment and all explosion protection provisions, if any.

Do not practice any working methods which may endanger safety

3.2 Qualified personnel

The probe may only be installed, operated and serviced by authorized personnel with special skills in measuring technology and in strict compliance with these operating instructions as well as the valid provisions.

The failure to observe these instructions – no matter whether intentionally or negligently – releases Pfaudler Werke GmbH from all liability and warranty claims. 4 Construction and type designation

4.1 Structural designs

The pH probe is available in two types of construction:

- Rod probe
- Ring probe

4.2 Type designations of the rod probe

The rod probe design is available in two different types

- Type 03
- Type 04

These types differ by the structure of their diaphragms.

Type 03 uses a ground, shrunk-in glass puck as part of the diaphragm.

Type 04 has a ground, screw-in glassteel puck as part of the diaphragm.

These pH probes are approved for use in zone 0. (refer to Annex 2)

EX pH rod probes type 03 delivered prior to July 2004 are <u>not</u> approved for use in zone 0. The data indicated on the nameplate is mandatory in this respect.

These two types are manufactured in two different designs which differ by their electrolyte reservoirs.

Rod probe in design K

In the present operating instructions, this design will also be referred to as **rod probe K**. The electrolyte reservoir of this probe type is permanently attached to the probe head (refer to Fig. 1).

Rod probe in design N

In the present operating instructions, this design will also be referred to as **rod probe N**. For this design, the electrolyte reservoir is supplied as a spare part which is installed separately next to the probe on location. The electrolyte reservoir is linked to the probe head by a hose included in the delivery (refer to Fig. 1).

In summary, the following should be remembered: There are four different types of rod probe which are named as follows:

- 1. Type 03/K
- 2. Type 03/N
- 3. Type 04/K
- 4. Type 04/N

For the available sizes, please refer to tables 2 and 3 in Section 9.1.

4.3 Type designations of the rod probe in dual design

Two independent pH values can be measured using one dual pH rod probe, e.g., for redundant applications. As the same design types apply to "dual probes" and for "single probes", they have the same type designation, only the term "dual" is added. For the available sizes, please refer to tables 2 and 3.

4.4 Type designations of ring probes

The ring probe is only available as type 03. It is equipped with a ground glass puck as part of the diaphragm.

The ring pH probe is approved for use in zone 0. (refer to Annex 2)

$\langle \xi_x \rangle$ Ring pH probes delivered prior to July 2004 are <u>not</u> approved for use in zone 0. The data indicated on the nameplate is mandatory in this respect.

Ring probes are manufactured in design N only, i.e., the electrolyte reservoir is supplied as a separate part and installed separately next to the probe on location. The reservoir is linked to the ring probe by a hose included in the delivery. (refer to Fig. 1)

As there is one type and one design of the ring probe only, it is not specified in detail but rather generally referred to as ring probe. Standard ring, probes are available in size DN50 and DN80. Larger dimensions are available on request.



Fig. 1 Overview of design options

5 Technical description

5.1 Measuring principle

The pH value is a measure of the hydrogen ion activity aH+ in a solution. The pH value is defined as the negative logarithm of the hydrogen ion activity a_{H+} . Of practical importance is the pH value in the range between pH 0 and pH 14. That is, pH 0 is a strong acid, pH 14 is a strong alkali, and pH 7 is neutral.

Glassteel pH probes made by Pfaudler operate according to the principle of potentiometry, i.e. a pH-dependent potential is built up at the pH probe that is subject to the hydrogen ion activity in an aqueous solution. This potential is derived and processed by a pH transmitter.

The actual sensor of our pH probes consists of pH-sensitive glass. At the phase limit between the pH glass and the measured solution, the so-called gel layer, an ion exchange (hydroxilation) takes place in the course of which alkali ions of the pH glass are replaced with hydrogen ions of the measured solution. The thickness of the gel layer is approx. 5 - 150 nm. The pH-dependent potential building up in the gel layer is transferred by a ionic conductor to a metal derivative layer located below the pH glass.

The pH sensor is a phase boundary structure between two chemically distinct substances with a liquid phase and a solid phase. Such a phase boundary structure is referred to as a half-cell. The potential of a half-cell, which is also called phase boundary or Galvani potential, cannot be measured directly because a measuring instrument would constitute another phase boundary in the measuring loop whose potential in turn would be unknown. For this reason, the potential of a half-cell can only be determined by a series connection of a second half-cell. The "liquid phases" of the half-cells are connected via a diaphragm. The second half-cell, the so-called reference electrode, supplies a defined, constant potential due to its very construction. The series connection of the pH electrode and the reference electrode provides a measuring signal that consists of the sum of the variable "pH potential" and the constant "reference potential". The measured signal is supplied to a pH transmitter for evaluation through appropriate connections.

5.2 Construction and function of the rod probes

pH rod probes made of glassteel consist of the following two principal components:

Measuring probe

Reference system

The measuring probe consists of a glasslined steel element containing the measuring electrode and the diaphragm. The reference system consists of the electrolyte reservoir with the reference electrode inside. These two components are linked to each other by the so-called electrolyte line. The individual components will be described in detail below in this chapter.

For the rod probes, a steel tube with a welded-on flange is used as sensor. The tube and the bottom of the flange are continuously coated with glass. The probe head and the terminal box are attached to the upper end of the flange.

The pH-sensitive glass, which will also be referred to as **"pH glass"** in this manual, has been fused onto a metal derivative layer at the end of the tube. Depending on the probe size, the sensor area is 120 to 350 cm².

The potential is derived from the derivative layer connected to the terminal box on the probe flange by a metal strip fused into the glass. The derivative layer and the metal strip are embedded in the glass, which provides a high-resistance insulation with respect to the steel and the surface. The metal strip used to derive the potential is connected to a special terminal socket in the terminal box which relays the measured signal through the Pfaudler connection cord (refer to section 9.10) to the transmitter or the isolation amplifier.

The combination of the pH glass and the metal derivative layer results in the pH-sensitive measuring electrode.

The diaphragm, which is also called a ground or split diaphragm, is located at the tip of the probe tube. The diaphragm has the task of making a conductive connection between the reference system and the product while preventing the product and the electrolyte from mixing.

The diaphragm of type 03 consists of a ground glass puck and the probe tube end which is glass-coated and ground inside. The glass puck is shrink-fitted into the end of the probe tube. The contact area between the glass puck and the probe tube forms the diaphragm gap.

The diaphragm of **type 04** consists of a ground glassteel puck with a threaded bolt, the so-called diaphragm bolt, and the glasslined, ground ring area at the face end of the probe tube. The diaphragm bolt is turned into the face of the tube. The contact area between the diaphragm bolt and the probe tube forms the diaphragm gap.

On the outside, the diaphragm is in contact with the measured solution (product). As the diaphragm gap allows penetration of electrolyte, the electrolyte line provides a conductive link between the reference system and the product.

The electrolyte line consists of a thin plastic hose leading from the probe head to the diaphragm. This plastic hose is routed through a stainless steel tube inside the probe. The electrolyte line can be vented through a venting plug at the probe head. The electrolyte reservoir of rod probe \mathbf{K} (K reservoir) is permanently attached to the probe head. The electrolyte reservoir of rod probe \mathbf{N} (N reservoir) is installed separately on location next to the probe and linked to the probe head via a PP hose included in the delivery.

The reference electrode is installed inside the electrolyte reservoir on location.

The electrolyte reservoir must be permanently pressurized with compressed air. This is to ensure that electrolyte can permanently reach the diaphragm and finally the product through the electrolyte line. The permanent electrolyte flow is designed to avoid contamination of the diaphragm. Furthermore, it prevents product from entering the electrolyte line through the diaphragm gap and clogging the electrolyte line.

The combination of the reference electrode and the measuring electrode and their conductive connection through the electrolyte line, diaphragm and product forms the measuring chain

When the probe is immersed in the product, the measuring chain supplies a highresistance, pH-dependent potential that is converted by a suitable pH transmitter into a standard signal in proportion to the pH value.

The measured pH is influenced by the product temperature because the slope of a measuring chain depends on the temperature according to Nernst's constant (refer to Sect. 6.1). The rod probes are equipped with a Pt100 for measuring the product temperature. Thus, the temperature-dependent portion of the pH change in the transmitter is automatically compensated.



Fig. 2 Components of rod probes

5.3 Construction and function of the dual rod probes

The dual rod pH probes are identical to the single-rod pH probes in terms of construction, however, they have two separate measuring systems comprising one measuring electrode, one Pt100 and one connection terminal each. As a standard, the dual probe is supplied together with an N reservoir and two reference electrodes. If necessary, two isolation amplifiers may be attached to the electrolyte reservoir. In order to obtain two independent measuring loops, two pH transmitters or one dual transmitter must be connected to the system. The connection to the pH transmitters is made by two Pfaudler connection cords (refer to Sect. 9.10). The functional principle of "dual probes" is identical to that of "single probes".

5.4 Construction and function of the ring probes

Ring pH probes made of glassteel consist of the following two principal components:

- Measuring probe
- Reference system

The measuring probe consists of a glasslined steel element containing the measuring electrode and the diaphragm. The reference system consists of the electrolyte reservoir and the reference electrode inside. These two components are linked to each other by the so-called electrolyte line. The individual components will be described in detail below in this chapter.

The sensor of a ring probe consists of a glasslined steel ring to the inside of which the pH-sensitive glass, which will also be referred to as "**pH glass**" in this manual, has been fused onto a metal derivative layer along a 300° perimeter. The sensor's surface area is approx. 30 cm² in size DN50 and approx. 50 cm² in size DN80. The probe head and the terminal box are attached to the side of the ring.

The potential is derived from the derivative layer connected to the terminal box by a metal strip fused into the glass. The derivative layer and the metal strip are embedded in the glass, which provides a high-resistance insulation with respect to the steel and the surface. The metal strip used to derive the potential is connected to a special terminal socket in the terminal box which relays the measured signal through the Pfaudler connection cord (refer to section 9.10) to the pH transmitter or the isolation amplifier.

The combination of the pH glass and the metal derivative layer results in the pH-sensitive measuring electrode.

The diaphragm, which is also called a ground or split diaphragm, is located at the inside surface of the intermediate ring. The diaphragm has the task of providing a conductive connection between the reference system and the product while preventing the product and the electrolyte from mixing. The diaphragm consists of a ground glass puck and a glasslined, ground drilling at the inside of the ring probe. The glass puck is shrink-fitted into the drilling. The contact area between the glass puck and the drilling forms the diaphragm gap.

On the outside, the diaphragm is in contact with the measured solution (product). As the diaphragm gap allows penetration of electrolyte, the electrolyte line provides a conductive link between the reference system and the product.

The electrolyte line consists of a thin plastic hose leading from the probe head to the diaphragm. This plastic hose is routed through a drilling inside the probe. The electrolyte line can be vented through a venting plug at the probe head.

The electrolyte reservoir of the ring probe (N reservoir) is installed separately on location next to the probe and linked to the probe head via a PP hose included in the delivery.

The reference electrode is installed inside the electrolyte reservoir on location.

The electrolyte reservoir must be permanently pressurized with compressed air. This is to ensure that electrolyte can permanently reach the diaphragm and finally the product through the electrolyte line. The permanent electrolyte flow is designed to avoid contamination of the diaphragm. Furthermore, it prevents product from entering the electrolyte line through the diaphragm gap and clogging the electrolyte line.

The combination of the reference electrode and the measuring electrode and their conductive connection through the electrolyte line, diaphragm and product forms the measuring chain. When the probe is covered with product, the measuring chain supplies a high-resistance, pH-dependent potential that is converted by a suitable pH transmitter into a standard signal in proportion to the pH value.

The measured pH is influenced by the product temperature because the slope of a measuring chain depends on the temperature according to Nernst's constant (refer to Sect. 6.1). The ring probes are equipped with a Pt100 for measuring the product temperature. Thus, it is possible to automatically compensate the temperature-dependent portion of the pH change in the transmitter.



Fig. 3 Components of the ring probe

5.5 Reference electrodes

Two different types of reference electrodes are available for the pH probes described in this manual:

- Silver acetate electrodes, letter symbol: AgAGel
- Silver chloride electrodes, letter symbol: AgAgCl

These reference electrodes differ in terms of potential and useful life. The zero point of the probes is decisively influenced by the potential of the reference electrode (refer to Sect. 6.2).

The measuring chain zero when using silver acetate electrodes is reached at approx. pH 1.35 \pm 1 (refer to Sect. 6.2). The isotherm point of our pH probes is also reached at pH 1.35 ±1 (refer to Sect. 6.3). As the pH values of these two characteristic values are identical, commercially available pH transmitters of the type normally used for glass electrodes may be combined with silver acetate electrodes. A single value has to be parameterized as zero point and isotherm point with these pH transmitters only. It is important, however, that the zero point can be set in the range of pH 1.35 ± 1 at the transmitter.

A silver acetate electrode has a useful life of 12 months from its delivery, regardless of whether KCl or K_2SO_4 is used as electrolyte.

The measuring chain zero when using silver chloride electrodes is reached at approx. pH 8.65 \pm 1 pH (refer to Sect. 6.2). Therefore, the pH values of the zero point and the isotherm point are not identical. The transmitter selected for use with silver chloride electrodes should offer independent, free parameterization of the zero point and the isotherm point.

A silver chloride electrode has a minimum useful life of 24 months from its delivery if KCl is used as electrolyte. If K_2SO_4 is used, its life is reduced to 12 months from the date of delivery. Upon delivery, the reference electrodes are protected by special envelopes against drying out and damages. The reference electrodes must be installed within 6 months of delivery in order to avoid premature fatigue.

For more details concerning the reference electrode, please refer to Section 11.4.

5.6 Electrolyte reservoir

Two different types of electrolyte reservoir are available for the probes described in this manual:

Electrolyte reservoir in design **"K"** In the present operating instructions, this design will also be referred to as **K reservoir**.

Electrolyte reservoir in design **"N"** In the present operating instructions, this design will also be referred to as **N reservoir**.

Main features of the K reservoir

The K reservoir has a capacity of 0.7 liters and can only be used in combination with rod probes. The reservoir is directly attached to the probe head. A T-piece is screwed into the upper end of the reservoir. The compressed air is connected to the lateral port of the T-piece. The compressed air cannot be shut off at the reservoir, therefore, we recommend fitting a shut-off valve to the air line. The upper port of the T-piece is used to fill the reservoir and to install the reference electrode. For this reason, the reference electrode first has to be removed before electrolyte can be refilled. The isolation amplifier cannot be attached directly to the reservoir but rather has to be installed separately as close as possible to the probe. The K reservoir is approved for a max. internal pressure of 10 bar. A higher internal pressure is not permitted.

Main features of the N reservoir

The N reservoir has a capacity of 1.7 liters and can be used in combination with rod probes or with ring probes. The reservoir is installed separately next to the probe and linked to the probe via a PP hose included with the delivery. A shut-off valve for compressed air connection is available on the cover of the reservoir. Reservoirs delivered in 2003 or later are equipped with an additional non-return valve in the shutoff valve. There are three tapped holes in the mounting plate, i.e. one for filling the reservoir with electrolyte, and two for installing reference electrodes. (A second reference electrode is necessary for dual probes only.) If necessary, the electrolyte supply to the probe can be shut off by a ball valve attached to the bottom end of the reservoir. A maximum of two isolation amplifiers may be attached to the electrolyte reservoir. The N reservoir is approved for a max. internal pressure of 10 bar. A design with a max. internal pressure of 16 bar is available as a special option. The data indicated on the nameplate is mandatory with respect to the maximum allowable internal pressure.

5.7 Electrolyte

Two different types of electrolyte are available for the probes described in this manual:

- Potassium chloride **KCI**
- Potassium sulphate K₂SO₄

Based on our long-term experience we know that KCl is the optimum electrolyte for our probes. For this reason, we always recommend using KCl as a standard. Only if KCl is inappropriate, e.g., if no chloride may enter the product or if KCl reacts with the product, it is also possible to use K_2SO_4 .

As a standard, the electrolytes may be used in a temperature range between -5 and +140 °C. Within these limits, the electrolytes may remain in the reservoir and the probe. However, the pH can no longer be measured at frost temperatures because measurement becomes too slow. On request, the electrolytes are also available with additives which enable the probes to remain inside the reactor or the pipe down to a temperature of -30 °C.

5.8 Technical data Slope of the measuring chain:	57.2 ± 2 mV/pH at 25 °C
Zero point of the measuring chain:	$1.35\pm1~\mathrm{pH}$
Potential of the measuring chain, reference electrode made of silver acetate:	+200 to -800 mV
Potential of the measuring chain, reference electrode made of silver chloride:	+600 to -400 mV
lsotherm point:	at 1.35 \pm 1 pH
Internal resistance of the measuring chain:	$10^{8} - 10^{9} \Omega$ at 25 °C
Diaphragm resistance:	approx. 50 k $\mathbf{\Omega}$
Insulation resistance:	$\geq 10^{12} \Omega$
Internal capacitance (with connection cord):	≤ 10 nF
internal inductance (with connection cord):	negligible
Allowable working temperature:	0-140 °C
Allowable working pressure:	 -1/+9 bar or -1/+15 bar (The data indicated on the nameplate is mandatory)
Max. ambient temperature:	50 °C
Thermal shock resistance ΔT :	120 °C
Temperature measurement:	using Pt100
Resistance:	refer to Figure 5
Electrical data if used in potentially explosive atmosp Required type of protection for category 2 equipment	heres: EEx ia IIC
Power and signal circuits	For connection to certified, intrinsically safe electrical circuit only. L _i negligible C _i negligible
Required type of protection for category 1/2 equipment	EEx ia IIB
Power and signal circuits	For connection to certified, intrinsically safe electrical circuit only. The following maximum values apply to the measuring circuits of probes on a common probe carrier: $U_i = 30 V$ $\Sigma I_i = 100 \text{ mA}$ $L_i \text{ (probe) negligible}$ $C_i \text{ (probe) negligible}$ $C_{\text{connection cord}} \le 10 \text{ nF}$
	The maximum allowable inductance of all inductors in the supply circuits is 11 mH. The maximum allowable capacitance of all capacitors in the supply circuits is 180 nF.
EC type examination:	PTB 03 ATEX 2207 X

6 Technical characteristics of measurement

6.1 Slope of the measuring chain The slope S of a pH probe is defined as follows:

$$\mathbf{S} = \Delta \mathbf{E} / \Delta \mathbf{p} \mathbf{H}$$

- $\Delta \mathbf{E} = \text{change in potential of the} \\ \text{pH probe}$
- $\Delta \mathbf{pH} = \mathbf{pH}$ change of the measured solution

The theoretical slope of a pH probe is approx. 59.2 mV/pH at 25 °C. In practice, this value is not reached due to design and manufacturing restrictions. The slope of our glassed probe is approx. 57.5 mV/pH at 25 °C. In order to compare the slope of the probe with that of other probes, the value is always specified with respect to the standard temperature of 25 °C.

Due to the physical properties of the measuring electrode (pH glass), the slope of the probe is temperature-dependent. The slope changes according to Nernst's constant at a rate of 0.1983 mV per 1 °C and per 1 pH.

This temperature influence can be compensated by the modern pH transmitters used today. For this purpose, the transmitter must measure the temperature of the measured solution (product) and the most important characteristics of the probe – slope, zero point and isotherm point – must be programmed into the transmitter (parameterization) (refer to Sect. 10.5).

Each probe is tested and measured prior to delivery. The measured slope is specified in the test report supplied together with the probe (nominal value). This nominal slope value must be input into the transmitter in order to calculate and compensate the temperature-dependent pH value shift.

The slope is recalculated during each two-point calibration process. This new "current value" will then be used by the transmitter for the current pH measurement.

The slope of glassed pH probes remains constant during the entire life of the probe, i.e. the probes are not subject to aging. Regular recalibration of the glasslined pH probes is not necessary for this reason.

6.2 Zero point of the measuring chain

The measuring chain zero is the pH with a measuring chain voltage of zero (total potential). The total potential basically consists of the sum of the individual potentials of the measuring and reference electrode. The zero point is decisively influenced by the type of reference electrode, and thus the potential of the reference electrode.

Two different types of reference electrodes are available for the probes described in this manual (refer to Sect. 5.5):

- The measuring chain zero of a silver acetate electrode is reached at approx. pH 1,35 ±1 = **pH**₀.
- The measuring chain zero of a silver chloride electrode is reached at approx. pH 8,65 ±1 = **pH**₀

Each probe is tested and measured prior to delivery. The measured zero point is indicated in the test report supplied together with the probe (nominal value). This nominal zero point value must be input into the transmitter in order to calculate and compensate the temperaturedependent pH value shift.

The zero point is recalculated during each calibration process. The new "current value" will then be used by the transmitter for the current pH measurement.

When entering the parameters into the transmitter, it must be ensured that the programmed zero point matches the reference electrode used.

6.3 Intersection of isotherms

If the total potential of a measuring chain is plotted as a function of the pH value of a measured solution (product) for a certain, constant temperature of the measured solution in a diagram, a straight line is obtained which is also referred to as "isotherm". The slope of the isotherm is temperature-dependent (refer to Sect.



Fig. 4 Isotherm intersection

6.1). Two isotherms determined at two different product temperatures each have a different slope (ascending gradient), and therefore a common intersection. The intersection of two isotherms is referred to as the isotherm intersection, or isotherm point in this manual.

The coordinates of the isotherm point in the isotherm diagram are referred to as \mathbf{pH}_{is} and \mathbf{U}_{is} .

The isotherm point of the pH probes described in this manual is reached at pH $1.35 \pm 1 = \mathbf{pH}_{is}$. This value remains constant during the whole life of the probe.

The value \boldsymbol{U}_{is} depends on the reference electrode used.

The isotherm point must be input into the transmitter in order to calculate and compensate the temperature-dependent pH value shift.

If a silver acetate electrode is used, the pH values of the isotherm point and the zero point are **identical**. For this reason, the isotherm point need not be programmed if a silver acetate electrode is used. Only the nominal value of the zero point specified in the test report supplied together with the probe must be entered in the transmitter.

If a silver chloride electrode is used, the pH values of the isotherm point and the zero point are **not identical**. For this reason, the isotherm point must be programmed if a silver chloride electrode is used. Depending on the transmitter used, one of the two coordinates \mathbf{U}_{is} or \mathbf{pH}_{is} must be entered as isotherm point. The nominal values of these coordinates are provided in the test report supplied together with the probe. If these values are not readily available, the following values may be used instead:

 $\mathbf{U}_{is} =$ 420 mV or $\mathbf{pH}_{is} =$ 1.35 pH

Some units also require the input of both values.

If higher demands are made on the accuracy of measurement, especially at higher temperatures, we recommend using the measured isotherm point for operation. The isotherm point can be determined in our factory (optionally, subject to payment).

6.4 Resistance of the measuring chain

Short response times depend on the lowest possible resistance of the measuring electrodes. That means, that the resistance of the measuring electrode must be lower than the insulation resistance between the measuring electrode and the probe body. This requirement is met by a relatively low-resistance pH glass in combination with a high-resistance protective glass layer. The glassteel probe body is grounded for dissipating electrical leakage fields.

Resistance at 25 °C

- Measuring electrode:
- $10^8 \cdot 10^9 \Omega$, depending on probe type Reference electrode: max. 200 kΩ
- Insulation: $\geq 10^{12} \Omega$

6.5 Diaphragm resistance

The resistance of the diaphragm amounts to an average 50 k Ω , with 200 k Ω representing the max. permissible value. The diaphragm has been ground to ensure the minimum permeability (leakage rate) of the electrolyte. Based on the overpressure in the electrolyte system, the danger of contaminating the diaphragm is very low, and the same applies to the risk of diffusion potentials building up at the diaphragm.

The influence of the diaphragm on the total potential, and thus the pH value, is extremely small or even zero.

6.6 Measuring variance

The measuring variance of ± 0.1 pH results from the specified tolerance of the measuring chain zero of approx. ± 1 pH with respect to the isotherm point. The accuracy of the measurement can even be improved by determining the isotherm point and compensating the difference in the transmitter. The isotherm point can be determined in our factory (optionally, subject to payment).

6.7 Alkali error

The allowable measuring range is in between the lowest pH value and pH 10. Above pH 10, the alkali error produces a deviation that increases with temperature and the N_{a+} concentration.

6.8 Time response to pH change

Electrochemical processes at the surface of the measuring electrode and the time constant of the measuring loop determine the time response of the probe to pH changes. Up to strong alkali values, the electrochemical equilibrium is reached very fast, so that the influence of the electrical components of the measuring loop on the response times is of greatest importance.

The display's response to sudden changes of the pH value generally does not affect the measuring process. The final value is normally reached after a few seconds. Only above pH 8 it takes approx. one minute to reach the final value. This delay, which is also common with glass electrodes, is caused by ion exchange processes at the electrode surface.

6.9 Time response to temperature changes

The potential of the pH probe should settle with the lowest hysteresis possible even in the case of product temperature changes during pH measurement.

Due to its very design, the measuring electrode responds faster than the Pt100 in the probe to temperature variations. The resulting maximum deviation is less than 0.1 pH at a temperature change of 1 °C/min or less. However, the exact isotherm point of the measuring chain must have been entered in the transmitter for this purpose. The isotherm point can be determined in our factory (optionally, subject to payment).

7 Application limits

7.1 Chemical resistance

Glass is considered to be resistant if erosion is less than 0.1 mm per year. This is an average value which is determined in experiments to DIN-ISO and represented in so-called ISO corrosion curves. The resistance of our pH probes depends on the yellow pH-sensitive glass (pH glass) because the resistance of this glass is lower than that of our Pfaudler standard glass. Exact determinations concerning the resistance cannot be made unless the application is known and corrosion tests were made. For more details, please refer to our Publication 206, "Pfaudler compound glassteel materials".

7.2 Temperature

The allowable product temperature depends on the pH. The maximum admissible product temperature is 140 °C. For the permissible temperature range, please refer to Fig. 6.

For product temperatures above 100 °C, the pressure in the electrolyte line must be increased by increasing the vapor pressure in order to avoid boiling of the electrolyte and prevent interrupting the electrolyte line by gas bubbles. Due to the rising resistance of the measuring electrode with falling temperatures and the associated delayed display response, the lower temperature limit is 0 °C. However, due to the composition of the electrolyte, the pH probes may remain inside the reactor down to a temperature of -5 °C. On request, special electrolytes are available which enable the probes to remain inside the reactor or the pipe up to a temperature of -30 °C. However, the pH can no longer be measured at frost temperatures.



Fig. 5 ISO corrosion curve of pH glass



Fig. 6 Application range of the pH probes

The glassed pH probe is resistant to thermal shock up to a ΔT of max. 120 °C (temperature difference between product and probe). After the first 5 - 10 tem-

perature changes, however, there may be a slight zero point drift which can be corrected by single-point calibration (refer to Section 10.10).

7.3 pH range

For the permissible pH application range, please refer to Fig. 6. Limitations are caused by the chemical resistance (cf. Section 7.1) and the alkali error (cf. Section 6.7). Prolonged uses in the high alkali range should be avoided because the probes would otherwise require a longer initial setting time in the neutral and acid range. In ranges below pH 8, the probe regenerates very quickly at temperatures above 50 °C.

7.4 Pressure

Probes with flanges up to and including DN150 are suitable for an operating pressure of -1 to +15 bar. Probes with flanges sized DN200 ore more are suitable for an operating pressure of -1 to +10 bar. A higher operating pressure is not permitted.

The electrolyte reservoirs are approved for a max. internal pressure of 10 bar as a standard. The N reservoir is also available as a special design with a max. internal pressure of 16 bar.

The data indicated on the nameplate is mandatory with respect to the allowable pressure level of an electrolyte reservoir. A higher internal pressure is not permitted.

During operation, the pressure inside the probe (= pressure inside the electrolyte reservoir) should be at least 1 bar above maximum possible operating pressure or the temperature-dependent vapor pressure of the probe. This is to ensure that product is prevented from entering the probe through the diaphragm gap and clogging the electrolyte system. Furthermore, it is ensured that the diaphragm gap is always in contact with electrolyte. A pressure regulation system is not necessary. Once set to the highest process pressure, the internal pressure of the probe does not have to be readjusted. The differential pressure may be greater than 1 bar to the maximum rated pressure, this will cause increased electrolyte consumption.

Examples of proper pressure settings:

1. Pressure inside the reactor:	4 bar max.
Vapor pressure:	1 bar at an operating temperature of 100 °C
Minimum internal pressure:	5 bar

2. Pressure inside the reactor: 2 bar max.
Vapor pressure: 4 bar at an operating temperature of 140 °C
Minimum internal pressure: 5 bar

If the probe is subject to process pressure inside the reactor or the pipeline, it must be ensured that the pressure inside the electrolyte system is permanently at least 1 bar above the process pressure. Otherwise, there is a risk of product entering the probe through the diaphragm gap and clogging the electrolyte line.

7.5 Inappropriate products

- Reliable pH measurements are not possible in water-free products (water content < 1 %).
- All fluorine-containing products below pH6 lead to strong corrosion.
- Strongly hygroscopic products and the application of dry nitrogen to an empty reactor or pipeline will drain the gel layer, leading to a loss of the slope, which can be corrected by reconditioning the pH probe (refer to Section 10.2).

8 pH transmitters and accessories for pH probes

8.1 List of pH transmitters approved by Pfaudler

Only suitable equipment may be used as pH transmitters which must have the following features:

- The unit must have a high-impedance input, $R_i \ge 10^{12} \Omega$
- It must be possible to set the zero point and the isotherm point valid for the probe.

The following units have been approved:

- Knick: 71(X) pH with option 356* 73 pH with option 356* 74 pH with option 356* 76(X) pH with option 356* 77(X) pH with option 356* PROTOS 3400(X) with module pH 32
- Siemens: Sipan 3 P Sipan 32 (X) Sipan 34
- Yokogawa: EXA pH 200 EXA pH 202 EXA pH 400 EXA pH 402 EXA xt pH 150 EXA xt pH 450
- E+H: Mypex CPM 340 Mykom CPM 121/151/ 152/221/252/431

Polymetron: Monec 9135

Rosemount: Model 54 pH

For programming the transmitters, please refer to the manufacturers' operating instructions.

* the nominal zero point and the nominal slope can be parameterized For probes immersed in product, the transmitter or the isolation amplifier must not be disconnected from mains for more than 1 - 2 hours. Otherwise, polarization may result in a zero drift which may eventually be outside the setting range of the pH transmitter. Where longer disconnection is necessary, it is important to disconnect the measuring circuit at the probe either by removing the plug from the reference electrode or at the probe's terminal box.

8.2 Isolation amplifier

For reasons of operational safety and to minimize interference, an isolation amplifier should be connected in series with the probe and the transmitter. The isolation amplifier ensures electrical isolation of the input and output as well as impedance transformation. The input impedance is approx. $10^{13} \Omega$. The output impedance is approx. 5 Ω . If the probes are used in reactors, apparatus or pipelines made of insulating materials, high potential differences may occur in the presence of solvents which do not allow for measurement without an isolation amplifier. If an isolation amplifier is ordered together with the probe, it will be attached to the N electrolyte reservoir for N probes already in the factory. For K probes, the isolation amplifier must be installed by the customer as close as possible to the probe. The isolation amplifier is supplied with power by an external power supply unit or a transmitter. All transmitters approved by Pfaudler can be connected to the low-impedance output of the isolation amplifier.

8.3 Power supply

Commercially available power supply units may be used for the isolation amplifier which are capable of supplying the necessary power of 8.5 V/10 mA. Where explosion protection is necessary, power supply units with an intrinsically safe output must be used. We recommend using the power supply unit type WG10A7 made by Knick.

8.4 Integrator

Interference pulses in the line between the isolation amplifier and the transmitter, which are caused by electric or magnetic fields or by quick and high pH changes in the product, may be eliminated or attenuated (full duration half maximum of approx. 2 s) by an RC integrator connected directly to the input of the transmitter. The integrator may be used in an intrinsically safe measuring circuit if the permissible external capacitance is not exceeded. The integrator capacitance is 0.33 μ F (Part no. 068 262-).

9 Installation and electrical connection of the probes

9.1 Installation of rod probe in the reactor

Before installing a rod probe in the reactor, you should verify whether there is sufficient distance to the agitator and the reactor wall. If necessary, suitable spacers or reducing flanges must be used.

The rod probe K may only be installed vertically, with the electrolyte reservoir on top, and in orientations that diverge by max. 45° from this position.

The rod probe N may be installed in any orientation because the overpressure in the electrolyte reservoir ensures at any time that electrolyte may flow from the electrolyte reservoir to the probe, regardless of the orientation of the probe.

Depending on the required length, rod probes are available with flange sizes DN50, DN80, DN100, DN150 and DN200. For nozzles with a nominal width in between these values, special designs of reducing flanges may be obtained.

For reactors not in compliance with DIN 28136, the probes are not always available in suitable lengths. In these cases, a longer probe must be installed with a spacer in order to achieve the optimum immersion length (refer to Tab. 3). If you are not sure, it is recommended to contact the manufacturer. Suitable spacers are available.

When using your own reducing flanges, please make sure that the element covering the contact area below the terminal box does not sit on the reducing flange (cf. Fig. 7). In the event of non-compliance, the glass may be damaged at the bottom side of the flange, and the fused-in metal strips may be interrupted.

Assembly process:

- Put down flange gasket.
- ▼ Remove electrode cap.
- ✓ Protect the nozzle and the probe against damage by a piece of cloth or a PTFE sleeve. Slowly introduce the probe into the reactor through the nozzle. Avoid pendulum motion.
- ▼ Tighten flange screws evenly crosswise with the prescribed tightening torque; (cf. Tab. 1).

Measuring probes type 04 must not be rested on the diaphragm bolt!



Fig. 7 Installation example with reducing flange

Table 1 Tightening torques of split flange connections
--

		Max. tightening torques in Nm with admissi operating pressures of:		
Flange	Screws	-1 to +10 bar	-1to +16 bar	
DN50	4 x M16	30	30	
DN80	8 x M16	35	35	
DN100	8 x M16	35	35	
DN150	8 x M20	40	40	
DN200	8 x M20	55		

Reactor type		Nozzle		Probe dimensions				
	DN Designation		Designation $\mathbf{D} \times \mathbf{I}_1$ \mathbf{I}_2			I ₃		
			_	_	К	N		
			[mm]	[mm]	[mm]	[mm]	[liters]	[%]
-	50	-	38 (46) x 300	140	485	200	-	-
AE 63	50	N8*	38 (46) x 450	140	485	200	55	87
AE 100	50	N8*	38 (46) x 670	140	485	200	68	68
AE 160	50	N2 N10	38 (46) x 670	140	485	200	100	62
AE 250	50	N2 N10	38 (46) x 800	140	485	200	137	55
AE 400	80	N2 N10	38 (46) x 950	140	485	200	214	53
AE 630	100	N2 N10	83 x 1150	180	500	215	258	41
AE 1000	100	N2 N3	83 x 1400	180	500	215	303	30
AE/BE 1600	100	N10	83 x 1600	180	500	215	454	28
CE 1600	100	N3	83 x 1400	180	500	215	477	30
AE/BE 2500	100	N2 N3	83 x 1800	180	500	215	688	27
CE 2500	100	N2 N3	83 x 1600	180	500	215	348	14
AE/BE 4000	150	N2 N3	83 x 2000	180	505	220	1345	33
CE 4000	150	N2 N3 N10	83 x 2000	180	505	220	859	21
AE/BE 6300	150	N10	127 x 2500	180	505	220	1762	28
CE 6300	150	N2 N3 N10	127 x 2500	180	505	220	1244	20
BE 8000 ø 2000	150	N2 N3 N10	127 x 3000	180	505	220	1432	18
CE 8000 ø 2000	150	N2 N3 N10	127 x 2850	180	505	220	1229	15
BE/CE 8000 ø 2200	150	N2 N3 N8 N10	127 x 2500	180	505	220	1689	21
BE/CE 10000	200	N2 N3 N8 N10	180 x 2700	180	515	230	1764	18
BE 12500	200	N2 N3 N6 N8 N10	180 x 3200	180	515	230	2154	17
CE 12500	200	N2 N3 N6 N8 N10	180 x 2700	180	515	230	4319	34
BE/CE 16000 ø 2600	200	N2 N3 N6 N8 N10	180 x 3200	180	515	230	3967	24
BE/CE 16000 ø 2800	200	N2 N6 N8 N10	180 x 3200	180	515	230	1944	12
BE/CE 20000	200	N2 N6 N8 N10	180 x 3200	180	515	230	6055	30

Table 2 Principal dimensions of the probes and allocation to DIN reactors



* Instead of a baffle

Nozzle design to DIN 28 139, flange design to DIN 28 150, connection dimensions to DIN 2501, min. PN10. The nozzle designations correspond to the catalog sheets.

Please insert special spacers when using the measuring probe in reactors with anchor-type agitators. In this case, please contact us.

Fig. 8 Principal dimensions of DIN reactors

Reactor type	Nozzle		Probe dimensions					Minim	Minimum volume	
	DN	Designation	D x I ₁	l ₂		l ₃	DN x I ₄			
					К	N				
			[mm]	[mm]	[mm]	[mm]	[mm]	[liters]	[%]	
L 160	50	Р	38(46) x 670	140	485	200	n/a	97	60	
DG 100	50	0	38(46) x 450	140	485	200	n/a	97	97	
DG 250	50	R	38(46) x 670	140	485	200	n/a	178	71	
DG 500	50	0 S	38(46) x 670	140	485	200	n/a	328	65	
DG 800	50	0 S	38(46) x 950	140	485	200	50/80 x 45**	528	66	
Т 100	50	P	38(46) x 450	140	485	200	50 x 70	78	78	
T 200	50	P	38(46) x 450	140	485	200	n/a	162	81	
T 300	50	P	38(46) x 670	140	485	200	50 x 30	171	57	
T 500	50	P	38(46) x 670	140	485	200	50 x 110	303	60	
T 800	50	P	38(46) x 950	140	485	200	50/80 x 45**	378	47	
E 1200	100	O R	83 x 1150	180	500	215	100 x 120	540	45	
E 2000	100	O R	83 x 1400	180	500	215	100 x 100	681	34	
E 3000	100	NORT	83 x 1600	180	500	215	100 x 100	886	29	
E 4000	100	NORT	83 x 1800	180	500	215	n/a	1200	30	
E 6000	100	NOT	83 x 2150	180	500	215	n/a	1732	29	
E 8000	100	NOST	83 x 2150	180	500	215	n/a	3533	44	
E 12500	150	PV	127 x 3000	180	505	220	n/a	1894	15	
E 16000	200	OU	180 x 3200	180	515	230	n/a	2951	18	
E 20000	200	OU	180 x 3200	180	515	230	n/a	5768	29	

Table 3 Principal dimensions of the probes and allocation to reactors according to Pfaudler standard



** Reducing flange in special design

Inclined nozzles for reactors T 100-T 800

Nozzle design to DIN 28 139, flange design to DIN 28 150, connection dimensions to DIN 2501, min. PN10. The nozzle designations correspond to the catalog sheets.

Please insert special spacers when using the measuring probe in reactors with anchor-type agitators. In this case, please contact us.

Fig. 9 Principal dimensions of reactors to Pfaudler standard

9.2 Installation of rod probe in a pipeline

The rod probe K may only be installed vertically, with the electrolyte reservoir on top, and in orientations that diverge by max. 45° from this position.

There are three installation options:



Fig. 10 Installation in a T-piece

1. Installation in a T-piece

When determining the necessary probe length and flange dimensions, the situation on location must be observed. The probe must be long enough to ensure that the pH glass is fully covered with product. The rod probe N may be installed in any orientation because the overpressure in the electrolyte reservoir ensures at any time that electrolyte may flow from the electrolyte reservoir to the probe, regardless of the orientation of the probe.



Fig. 11 Installation in a 90° elbow

2. Installation in a 90° elbow with an additional nozzle

When determining the necessary probe length and flange dimensions, the situation on location must be observed. The probe must be long enough to ensure that the pH glass is fully covered with product.

Fig. 12 Installation in installation fitting

3. Installation in a Pfaudler installation fitting made of glass fiber reinforced plastic (GFRP) with a flushing fitting The installation fitting is available with the specified dimensions as a standard, part no. 383 127-. Installation fittings with other dimensions are available on request.

For admissible tightening torques, refer to Table 1.



9.3 Installation of ring probe in a pipeline

The ring probe can be installed in any orientation but vertical installation is preferred.

For horizontal pipelines, please ensure that the pH glass and the diaphragm are completely covered with product during operation, otherwise, incorrect measurements may occur.

When installed in non-conductive pipelines, such as glasslined or plastic pipelines, conductive (black) gaskets must be used.

Due to the danger of element formation, non-conductive (white) gaskets must be used with conductive pipelines. For admissible tightening torques, refer to Table 1.

9.4 Installation of K reservoir

The reservoir is directly attached to the probe head already in the factory. Installation on location is not necessary.

It is not permitted to replace the plastic connection bolts with metal bolts. No metal parts are permitted anywhere in the area of the electrolyte! Only plastic lines may be used for the last 50 cm of the compressed air line.

Reason:

Electrolyte condensation may accumulate in metal pipelines which may form a conductive link with ground, thus causing false potential readings.

9.5 Installation of N reservoir

The N reservoir must be installed vertically and as close as possible to the rod probe or the ring probe, max. distance 10 m.

The electrolyte reservoir and the probe head are linked by a PP hose (delivered length 10 m). The hose fittings on the reservoir and the probe head allow for easy fastening of the connection hose without any tools.

It is not permitted to replace the plastic connection bolts with metal bolts. No metal parts are permitted anywhere in the area of the electrolyte! Only plastic hoses may be used for the last 50 cm of the compressed air line.

Reason:

Electrolyte condensation may accumulate in metal pipelines or bolted connections which may form a conductive link with ground, thus causing false potential readings.

If the electrolyte reservoir is installed in the open air or damp operating facilities, it may be appropriate to integrate the electrolyte reservoir and the electronic devices in a protective casing (option). Otherwise, moisture bridges or frost may disturb the measurement. When installed in the open air, a heater must be provided for the protective casing in order to avoid condensation at temperatures below the dew point.

The reference electrodes may fail at temperatures below zero.



Fig. 13 Terminal diagram for rod probe "K" with isolation amplifier



Fig. 14 Terminal diagram for rod probe "K" without isolation amplifier

9.6 Electrical connection of rod probe K

For the electrical connection of the probe to the transmitter, please refer to the circuit diagram and the operating instructions of the transmitter used. The circuit diagrams of the equipment approved by Pfaudler may be obtained from us.

A cable with a minimum area of 6 mm² must be connected between the ground terminal of the probe and the equipotential bonding point of the reactor or the pipeline using the shortest possible route.

The connection between the connector on the terminal box of the probe and the measuring transducer or the isolation amplifier is made using the Pfaudler connection cord (pH cable). Connecting the cables: Turn the connector into the right position in the socket and press until it is engaged. Open the PG11 spiral gland and turn the envelope fully onto the socket. Hold the envelope firmly and tighten the PG gland manually.

Both connections of the measuring chain of rod probe K are connected to the socket in the terminal box. Thus, the entire measuring signal and the Pt100 signal are transmitted by the pH cable to the transmitter or the isolation amplifier.

For the core assignment of the Pfaudler connection cord, please refer to Section 9.10.

In order to avoid any type of potential loss, please ensure that no moisture can enter the connector or the socket. If necessary, dry both parts with a hot-air dryer.

The connection between the isolation amplifier and the transmitter can be made using standard signal cables.



Fig. 15 Terminal diagram for rod probe "N" with isolation amplifier



Fig. 16 Terminal diagram for rod probe "N" without isolation amplifier

9.7 Electrical connection of rod probe N

For the electrical connection of the probe to the transmitter, please refer to the circuit diagram and the operating instructions of the transmitter used. The circuit diagrams for the equipment approved by Pfaudler may be obtained from us.

A cable with a minimum area of 6 mm² must be connected between the ground terminal of the probe and the equipotential bonding point of the reactor or the pipeline using the shortest possible route.

The connection between the connector on the terminal box of the probe and the measuring transducer or the isolation amplifier is made using the Pfaudler connection cord (pH cable). Connecting the cables: Turn the connector into the right position in the socket and press until it is engaged. Open the PG11 spiral gland and turn the envelope fully onto the socket. Hold the envelope firmly and tighten the PG gland manually.

The two connections of the measuring chain of rod probe N are designed separately. The measuring electrode is connected to the socket in the terminal box which relays the measured signal together with the Pt100 signal through the Pfaudler connection cord to the transmitter or the isolation amplifier.

The second part of the measured signal is connected to the free end of the reference electrode from where it is transmitted to the isolation amplifier or a transmitter using a special cable included in the delivery (reference electrode cable). The reference electrode cable is 1m long. If necessary, the cable may be extended by a singlecore, insulated line.

For the core assignment of the Pfaudler connection cord, please refer to Section 9.10.

In order to avoid any type of potential loss, please ensure that no moisture can enter the connector or the socket. If necessary, dry both parts with a hot-air dryer.

The connection between the isolation amplifier and the pH transmitter can be made using standard signal cables.



Fig. 17 Terminal diagram for ring probe with isolation amplifier



Fig. 18 Terminal diagram for ring probe without isolation amplifier

9.8 Electrical connection of the ring probe

For the electrical connection of the ring probe, the descriptions given in Section 9.7 apply analogously.



Fig. 19 Terminal diagram for isolation amplifier with rod probe K and Pt100 in 3-wire circuit





9.9 Electrical connection of isolation amplifier

The recommended isolation amplifier type 82G, Messrs. Knick, requires auxiliary power supply of 8.5 V/10 mA. Power supply can be provided in two different ways:

- Supply by a separate, suitable power supply unit. We recommend using the power supply unit type WG10A7 made by Knick.
- Supply by a power supply unit with sufficient power integrated in the transducer.

For the core assignment of the pH cable, please refer to Section 9.10.

9.10 Pfaudler connection cord

The Pfaudler connection cord (pH cable) is a prefabricated cable by which the proportionate pH measuring signal is transmitted from the probe to the transmitter or the isolation amplifier.

The cable is specifically designed for transmission of very high-impedance signals. As a standard, the cable is available in lengths of 3, 5 or 10 m.

A special connector is fitted to one cable end which fits into the socket in the terminal box of the probe. In order to protect the joint against moisture, the cable is equipped with an envelope with a double O-ring seal which is pushed over the connector and socket joint.

The other cable end has been stripped, and the individual cores are equipped with clamping terminals and numbered as shown in Fig. 21. For the assignment of cores, please refer to Table 4.



Fig. 21 Pfaudler connection cord (pH cable)

Core no.	1 (coax)	2 (coax)	3	4	5	6	8	9
Conductor color	white	brown	brown	green	blue	black	violet	orange
рН К		Reference electrode						
pH N		1)					Screen,	Screen,
pH ring	pH electrode	1)	Pt100	Pt100	Frame	Pt100	coaxial cable 1	coaxial cable 2
pH K dual	Reference electrode							
pH N dual		1)						

1) The potential of the reference electrode is relayed separately to the transmitter or the isolation amplifier using the reference electrode cable included in the delivery.

10 Start-up and calibration

10.1 Preliminary note

When the probe has been installed and connected as described in Section 9, start-up is basically carried out in four steps:

- Reconditioning of the probe (refer to Sect. 10.2)
- ▼ Filling the electrolyte reservoir, installation of the reference electrode and venting of the probe (refer to Sec. 10.3 - 10.4).
- Parameterization of the transmitter (refer to Sect. 10.5)
- ▼ Calibration of the probe (refer to Sect. 10.6 - 10.11).

The individual steps will be explained in detail below.

10.2 Reconditioning the probe

The probe must be reconditioned before it can be started up. By this process, a gel layer is built up on the surface of the pH glass. This gel layer is an important condition for a stable zero point of the measuring chain.

There are two ways of reconditioning the probe:

■ Watering of the probe for 24 hours.

- Or, if the reconditioning process is to be accelerated:
- Boiling the probe for one hour in slightly acidified water.

Reconditioning can be performed inside or outside the reactor or the pipeline.

10.3 K design: Filling, venting and installing the reference electrode

The permitted ambient temperature of the electrolyte reservoir is 0 to +50 °C. As a rule, the K reservoir should be filled only at a product temperature of less than 80 °C and without process pressure. If this is not possible, the filling process should not take longer than two minutes.

The electrolyte line must be free from bubbles in order to ensure a faultless electrical connection between the reference electrode and the diaphragm.

KCl is preferred as electrolyte, in special cases K_2SO_4 may also be used (refer to Sect. 5.7).

Only Pfaudler electrolyte may be used in order to guarantee the faultless function of the probes over a long period of time.

Process of filling and venting

- Remove the plug from the T piece on the upper side of the electrolyte reservoir. Fill in electrolyte through the opening up to the level mark (maximum level 0.7 liters).
- ▼ Screw the reference electrode provided into the filling port and plug in the reference electrode cable.
- Apply pressure to the electrolyte reservoir. The electrolyte line will fill with electrolyte.
- ✓ Plug a hose onto the venting plug and put the other end of the hose into a clean bottle. Open the venting plug by approx. one turn and leave it open until the electrolyte emerges without any bubbles. Then close the venting plug again.

Caution: If possible, open and close the venting plug only manually.

- Carefully wash off the remaining electrolyte in the area of the venting plug, the filling port and the screwed connection with water.
- Set the necessary operating pressure inside the electrolyte reservoir as described in Section 10.13 and check all details for leaks in order to avoid electrolyte bridges to the steel flange. Electrolyte bridges would falsify the measured result.

Do not vent the probe at a product temperature of more than 80 °C, otherwise, cold electrolyte may flow through the probe and cause a shock damage to the diaphragm.

10.4 N and ring design: Filling, venting and installation of the reference electrode

The permitted ambient temperature of the electrolyte reservoir is 0 to +50 °C.

The electrolyte line must be free from bubbles in order to ensure a faultless electrical connection between the reference electrode and the diaphragm.

KCl is preferred as electrolyte, in special cases K_2SO_4 may also be used (refer to Sect. 5.7).

Only Pfaudler electrolyte may be used in order to guarantee the faultless function of the probes over a long period of time.

Process of filling and venting

- Remove the closing plug from the filling port and fill in electrolyte through the opening up to the level mark (maximum level 1.7 liters). Screw the closing plug back in.
- Remove closing plug from one of the three installation openings, screw in the reference electrode and plug on the loose reference electrode cable supplied. (If a design with an isolation amplifier was chosen, the cable is already connected to the isolation amplifier)
- ✓ Apply pressure to the electrolyte reservoir. The electrolyte line will fill with electrolyte.
- ✓ Push a hose onto the venting plug and put the other end of the hose into a clean bottle. Open the venting plug by approx. one turn and leave it open until the electrolyte emerges without any bubbles. Then close the venting plug again.

Caution: If possible, open and close the venting plug only manually.

- Carefully wash off the remaining electrolyte in the area of the venting plug, the filling port and the screwed connection with water.
- Set the necessary operating pressure inside the electrolyte reservoir as described in Section 10.13 and check all details for leaks in order to avoid electrolyte bridges to the steel flange. Electrolyte bridges would falsify the measured result.

Do not vent the probe at a product temperature of more than 80°C, otherwise, cold electrolyte may flow through the probe and cause a shock damage to the diaphragm

10.5 Setting the pH transmitter parameters

The probe-specific characteristics, such as slope, zero point and isotherm point, as well as the user-specific settings must be entered in the transmitter (parameterized) before calibration can be carried out. The characteristic values are indicated in the test report supplied together with the probe. Some of the parameters to be entered are listed in Table 4. This list does not claim to be complete. For the parameters to be entered, please refer to the operating instructions of the transmitter used and the user's requirements. The parameters may be entered in parameter input mode or in calibration mode, depending on the transmitter used.

Table 5 Parameterization

Parameter	Value setting	Option selected
Slope	Enter value from test report	-
Zero point	Enter value from test report	-
lsotherm point	Enter value from test report (if silver chloride electrode is used) or same as zero point (if silver acetate electrode is used)	
Quantity to be displayed	-	рН
Temperature compensation	-	automatic
Temperature sensor	-	Pt100
Current output	0-20 mA or 4-20 mA	pH or mV

10.6 What is meant by calibration?

Calibration is defined as the adjustment of the transmitter to the characteristic of the pH probe.

The measuring chain of the pH probe supplies a potential that depends on the chemical properties and the temperature of the product. The transmitter measures this potential and calculates the current pH from the measured value. The potential of the measuring chain, and thus the pH, is influenced by various factors, such as the current potential of the reference electrode or inevitable specimen variance. These factors, some of which cannot be detected by their very nature, are largely eliminated by calibrating the probe.

10.7 Calibration methods

The following calibration methods are used in pH measurement technology:

- Calibration through data input at the transmitter (refer to Sect. 10.8)
- Two-point calibration (calibration using 2 buffer solutions of identical temperatures, refer to Sect. 10.9)
- Single-point or product calibration (calibration with product, refer to Sect. 10.10)
- Automatic calibration (refer to Sect. 10.11)

We recommend performing two-point calibration in the course of initial start-up and after prolonged service interruptions. The individual methods and their suitability for our pH probes will be described in the following sections.

10.8 Calibration by data input at the pH transmitter

This calibration method is used whenever a quick function test of the probe is to be performed and/or if no high measuring accuracy is required.

Process of calibration through data input

Depending on the transducer used, the calibration and/or parameterization mode must be activated and the characteristic values of the probe indicated in the test report must be entered. When the calibration mode has been exited, the probe is ready for use.

However, this calibration method does not guarantee faultless functioning or a defined accuracy of the probe. For this reason, we recommend performing a twopoint calibration as soon as possible after calibration by data input.

10.9 Two-point calibration

Two-point calibration is the calibration method that is used most frequently with our probes because the measuring accuracy achievable with this method is totally sufficient for most practical applications, and a good functional control of the pH measuring equipment is achieved.

For a two-point calibration, the pH value of two different buffer solutions is measured one by one at the same temperature. The measuring chain potential measured each time is then assigned to the relevant pH. Based on the two pH values and the measured product temperature, the slope and zero point values will be recalculated by the transmitter. Then, automatic temperature compensation is performed by the transmitter using these new, calculated values. Some preparations have to be made before the actual calibration can be started:

- A constant pH measurement requires a state of equilibrium at the diaphragm, i.e. an evenly moistened ground surface. For this purpose, the electrolyte reservoir must be pressurized approx. 1 hour prior to the beginning of the calibration.
- In order to reach the highest possible accuracy in the adjustment to the measuring chain characteristic, the pH values of the two buffer solutions should differ by at least 3 pH. We recommend using buffer solutions pH 3 and pH 7 for two-point calibration of our probes.

The first measurement must always be performed with the buffer solution whose pH is closest to the zero point.

- In order to avoid interference by static charges or detect possible insulation faults, the buffer solution must be grounded, in particular, if the calibration reservoir is made of plastic material. The buffer solution is grounded by connecting a wire between the solution and the equipotential bonding system.
- The probe can be calibrated inside or outside the reactor or the pipeline. The black protective cap included in the delivery may be used as calibration reservoir that holds the buffer solution. 250 ml of buffer solution are needed for the small probes with a diameter of 38 mm. The large probes with a diameter of 83 mm require 1000 ml of buffer solution. A telescopic rod with a screwed-on cup that takes up the calibration reservoir is available for calibrating the probe inside the reactor.

Process of two-point calibration

- Recondition the probe and rinse it with distilled water
- Immerse the probe in the first buffer solution, the yellow pH glass must be fully immersed. Remove bubbles at the diaphragm of type 03 by shaking the cup for a moment.
- Connect the buffer solution to ground potential
- ▼ Activate the calibration mode at the transmitter. Then select the *Two-point calibration* mode and enter the pH of the first buffer solution.
- ▼ Wait until the measured value displayed has stabilized and then start the calibration process. The measuring chain potential determined has now been assigned to the pH of the first buffer solution.
- ✓ When the first calibration step has been completed, rinse the measuring probe and the calibration reservoir with distilled water.
- ✓ Immerse the probe in the second buffer solution, the yellow pH glass must be fully immersed. The two buffer solutions must have the same temperature. Remove bubbles at the diaphragm of type 03 by shaking the cup for a moment.
- Connect the buffer solution to ground potential.
- ✓ Enter the pH value of the second buffer. Wait until the measured value displayed has stabilized and then start the calibration process. The measuring chain potential determined has now been assigned to the pH of the second buffer solution.
- ✓ When the second measurement has been completed, the ground wire must be removed from the buffer solution for testing the measuring equipment. The pH value must not change by more than ± 0.1 pH. In the event of greater discrepancies, an insulation fault in the measuring loop, in the reference loop (electrolyte reservoir) or at the cable might be the reason (refer to Sect. 11.11 – Troubleshooting).
- Exit calibration mode after successful calibration.

A two-point calibration should be carried out approx. one week after initial start-up and should be checked again by a singlepoint calibration after approx. 4 weeks (refer to Sect. 10.10).

10.10 Single-point calibration

Single-point or product calibration is performed in order to verify a pH measurement or for quality assurance purposes.

Process of single-point calibration

Take a product sample from the process using a suitable device. Then determine the pH value of this sample with the required degree of accuracy using a manual tester or in the lab. Compare the pH value thus determined to the value displayed on the transmitter. The value displayed can be corrected as follows in the event of minor deviations.

Activate the calibration mode at the transmitter. Then select the *Single-point calibration* mode and enter the pH measured manually. When you exit calibration mode, the "old" pH is replaced with the "new", correct pH.

In the event of major deviations, we recommend performing a two-point calibration or checking the entire pH measuring equipment.

10.11 Automatic calibration

Various pH transmitters offer automatic calibration. Since this mode has been specifically designed for the properties of pH glass probes, Pfaudler pH probes with a measuring electrode made of pH-sensitive glass cannot be calibrated using this mode.

10.12 Pressure setting

For more details concerning the proper pressure setting, please refer to Section 7.4.

If the probe is subject to process pressure inside the reactor or the pipeline, it must be ensured that the pressure inside the electrolyte system is permanently at least 1 bar above the process pressure. Otherwise, there is a risk of product entering the probe through the diaphragm gap and clogging the electrolyte line.

11 Operation and maintenance

11.1 Recalibration

Since glasslined pH probes are not subject to aging, recalibration is actually necessary only if calibration intervals have been prescribed, if the potential of the reference electrode has changed, or if a measuring error is suspected. Nevertheless, we recommend recalibrating the probe approx. every 6 months if it is used in normal operation. When recalibrating the probe, you reach a good reproducibility of the measured value if you perform a single-point calibration with the product to be measured. For a description of the single-point calibration process, please refer to Section 10.10.

11.2 Refilling the electrolyte reservoir

In order to ensure faultless operation of the measuring probe, the electrolyte reservoir must always be filled with a sufficient quantity of electrolyte. The end of the reference electrode must always be immersed in the electrolyte, otherwise, the measuring loop will be interrupted.

Only Pfaudler electrolyte may be used in order to guarantee the faultless function of the probes for a long period of time.

If a processor-based transmitter is used, activate the calibration mode and then the *HOLD* function before filling.

Procedure of filling a K reservoir

As a rule, a K reservoir should only be filled with electrolyte at a product temperature of less than 80 °C and without process pressure. If this is not possible, the filling process should not take longer than two minutes.

- ▼ Switch off the air supply.
- Remove the connection cord from the reference electrode.
- Carefully unscrew the reference electrode because the electrolyte reservoir is still pressurized, and the excess pressure first has to escape.
- ▼ Fill the electrolyte reservoir with electrolyte through the opening for the reference electrode up to the level mark (maximum level 0.7 liters).

To restart the unit, proceed in reverse order. Do not forget to deactivate the calibration mode!

Procedure of filling an N reservoir

Switch off the air supply.

- Close the electrolyte valve at the bottom of the electrolyte reservoir.
- Carefully turn out the filling plug on top of the electrolyte reservoir because the reservoir is still pressurized, and the excess pressure first has to escape.
- ▼ Fill the electrolyte reservoir with electrolyte through the filling hole up to the level mark (maximum level 1.7 liters).

To restart the unit, proceed in reverse order. Do not forget to deactivate the calibration mode!

11.3 Electrolyte consumption

At a differential pressure of one bar, a consumption of 0.1 to 0.2 ml/h is to be expected. If a higher differential pressure is present, the consumption may rise to 5 ml/h. We recommend contacting our measuring technology department if a higher consumption is noticed.

11.4 Inspection of the reference electrode

A silver acetate electrode has a useful life of approx. 12 months (refer to Sect. 5.5).

The useful life of a silver chloride electrode is approx. 24 months if KCl is used as electrolyte and approx. 12 months if K_2SO_4 is used as electrolyte (refer to Sect. 5.5).

This presupposes that the electrodes are permanently immersed in the electrolyte. After that time, the reference electrode must be replaced in order to avoid a zero drift of the measuring chain, and thus false measured values.

If you suspect that a reference electrode may be exhausted, there are two ways of checking this:

- Perform a pH measurement in a buffer solution or a product with a constant pH using the reference electrode. Activate the *mV* display mode on the transmitter and read the voltage returned by the measuring chain. Then replace the old reference electrode with a new one and read the voltage now returned by the measuring chain. If the difference between the two values is more than 20 mV, the old reference electrode can no longer be used.
- Measure the reference electrode to be tested against a new reference electrode of the same type, i.e. determine the voltage difference between the two electrodes. For this differential measurement, both electrodes must be immersed in the electrolyte. If the voltage difference measured exceeds 20 mV, the old reference electrode can no longer be used.

11.5 Glass testing The customary high-voltage spark test of the glassed surface is not permitted for glassed pH probes!

Before performing a high-voltage spark test on the reactor, the probe must be protected against damage by electrical or mechanical influences.

The glass of the pH probes can be tested for corrosion using the Pfaudler glass testing equipment of the types **Corrosion Detector, PA3OW, PMD** or **Glass Portector**. When monitoring a reactor with one of these glass testers, the integrated pH probes are also monitored automatically. In the event of a defect (corrosion or pore) in the pH glass, the pH measurement will fail because a defect produces a short-circuit in the measuring electrode.

Depending on the conductivity of the product, the accuracy of the pH measurement is influenced by a glass tester. For this reason, it must be ensured that the pH measurement and glass testing functions do not operate at the same time. Glass monitoring must be switched off during pH measurement.

11.6 Cleaning and sterilization of the probes

Glass is largely insensitive to contamination. It will be sufficient to check the pH glass and the area of the diaphragm for product residues. When you observe measuring errors or a slow setting behavior, the probe must be cleaned. In most cases, contaminated areas can be cleaned with water, a solvent or a liquid, non-abrasive detergent for stainless steel products. Do not use any metallic or abrasive materials. When cleaning the probes manually inside or outside the reactor, a 5-20 % acid, such as HCI may be used to remove deposits or product films, if this cleaning process is carried out at ambient temperature. Do not use any fluorine-containing acids because they strongly attack glass.

When handling acids, please make sure to observe the accident prevention regulations applicable to hazardous substances and their use.

The probe may also be cleaned inside the reactor or the pipeline by the so-called CIP method (cleaning-in-place).

Admissible CIP cleaning processes:

- 1.5-2 % alkaline solution, max. 85 °C, max. 1 h
- 1.5 % acid (HNO3), 60 °C, max. 15 min.
- Steam 134 °C, max. 2 h.

For CIP cleaning it must be ensured that the admissible alkali and acid concentrations as well as the maximum temperature or cleaning time are not exceeded. Otherwise, the pH glass would be subject to increased corrosion.

Caution: Corrosion doubles with every 10 $^{\circ}\text{C}$ of a temperature increase when using alkalis for cleaning.

Depending on the CIP method used, a zero drift may occur that is compensated after a prolonged period of time only. A zero drift can be corrected in an instant by reconditioning the pH probe (refer to Sect. 10.2).

The following media are permitted for sterilizing the probe:

- Product
- Water vapor
- Alcohol solutions
- Aseptic solutions

11.7 Cleaning the diaphragm of type 04

The diaphragm bolt of probe type 04 may also be unscrewed for cleaning. In order to avoid diffusion errors at the diaphragm, all traces of electrolyte must be removed from the inner space of the diaphragm. This error is characterized by a slow setting behavior or an unsatisfactory reproducibility of the measured values. We recommend cleaning probes with a small diaphragm (38 mm in diameter) every six months and probes with a large diaphragm (83 mm in diameter) once per year.

A special tool is required for loosening and tightening the diaphragm bolt which can be obtained from us:

- Part no. 591 293 for measuring probe diameter 38 mm
- Part no. 591 294 for measuring probe diameter 83 mm

The tightening torque of the diaphragm bolt is:

- **10 Nm** for probe diameter 38 mm
- **27 Nm** for probe diameter 83 mm



Figure 22 Assembly plan of N design

11.8 Assembly instructions for electrolyte hose kit of rod probe N

In the event of potential loss due to leaks in the probe head, or if the electrolyte line is clogged, the electrolyte hose must be replaced. Electrolyte hose kits are available in four lengths for this purpose, refer to Table 6.

An electrolyte hose kit consists of a PTFE hose (with an integrated thread) to which all necessary gasket elements have already been fitted in the right order.

Table 6	Part numbers	electrolyte	hose kit
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Probe length [mm]	Electrolyte hose kit Part no.
300	591 8931
450 to 1000	591 8942
1100 to 2150	591 8953
more than 2150	591 8964

Removal and assembly instructions (for item numbers refer to Figure 22)

- Close the ball valve (4) at the bottom of the electrolyte reservoir and open the venting plug (5) at the probe head by approx. one turn. It is not necessary to loosen the connection hose between the electrolyte reservoir and the probe head.
- Remove the probe head screws (2) and take off the pressure flange (1).
- Pull off the probe head (3) and pull the old electrolyte hose kit including all gasket elements out of the probe head using a set of pointed pliers (refer to detail Y above).
- Remove venting plug (5), clean the opening in the probe head and the venting plug with water and dry all parts.

 Unscrew and remove the diaphragm bolt (18) of probe type 04 using the appropriate assembly tool. Clean the diaphragm area and the diaphragm bolt with water and dry these parts.

Probe	Part no.
diameter	of assembly tool
38 mm	591 293-
83 mm	591 294-

- Check the diaphragm bolt and the ground surfaces at the probe tube and at the diaphragm bolt for integrity.
- Replace the diaphragm bolt if damaged. If damage is detected on the probe tube, the probe must be returned to the factory for repair.
- Screw in the diaphragm bolt manually and tighten it using a dynamometric key (part no. K01 338-). Pass the point with the clicking sound twice or three times.

Probe	Tightening
diameter	torque
38 mm	10 Nm
83 mm	27 Nm

- ✓ Only the outside of the diaphragm area of probe type 03 can be cleaned because the glass puck (17) is shrunkfit
- Clean the electrolyte tube (12) at the upper end of the probe head with water and dry these parts.
- Check O-rings (13) and spacing ring (14) in the probe head for proper fit and insert the spacer (15) in the probe head, refer to detail Y below.
- ✓ Slightly lubricate the electrolyte tube with silicone grease or vaseline, place the probe head carefully on the electrolyte tube and turn it slightly while pushing it onto the O-rings. Turn the probe head until the venting plug has reached its proper position (refer to view A).
- ✓ Cut the new electrolyte hose kit to proper length (supplied in excessive lengths). Use the old electrolyte hose kit as a guide to determine the length. Cut the PTFE hose with a sharp knife at an angle (refer to detail X) on both sides and make sure not to damage the thread (10). Cut the thread until it protrudes 4 - 5 cm from the end of the hose. It is important to cut the hose at an angle to ensure that the electrolyte can flow out of the hose easily.
- Lubricate the gasket elements (6) (9) on the new electrolyte hose kit with silicone grease or vaseline.
- ▼ Wipe the PTFE hose clean and push it fully into the probe through the opening at the probe head. If the hose is too long, it must be shortened. Otherwise, it might get bent at the slanted end and prevent or obstruct electrolyte flow (refer to detail X).

- ▼ Screw venting plug into the probe head.
- ▼ Place the pressure flange in position and tighten the bolts crosswise.
- ✓ Open the ball valve at the bottom of the electrolyte reservoir and the venting plug at the probe head. Close the venting plug again as soon as the electrolyte emerges without bubbles. The probe has now been vented and is fully operative again. For venting, the pressure should be at least 2-3 bar. Prior to venting, please make sure that there is enough electrolyte in the electrolyte reservoir. The electrolyte emerging while venting can be collected and reused, provided that it is collected in a clean reservoir.



11.9 Assembly instructions for electrolyte hose kit of rod probe K

In the event of potential loss due to leaks in the probe head, or if the electrolyte line is clogged, the electrolyte hose must be replaced. Electrolyte hose kits are available in four lengths for this purpose (refer to Table 6).

An electrolyte hose kit consists of a PTFE hose (with an integrated thread) to which all necessary sealing elements have already been fitted in the right order.

Removal and assembly instructions

(for item numbers refer to Figure 23)

- Open the venting plug (9) by approx. one turn and let the electrolyte flow out of the electrolyte reservoir. The electrolyte can be collected and reused, provided that it is collected in a clean reservoir.
- Shut off the compressed air supply and separate the air hose from the T piece (3).
- ✓ Pull off the connector of the reference electrode cable (1) from the reference electrode (2).
- ✓ Open the terminal box (10), disconnect the reference electrode cable from terminal 2 and pull the cable out of the screwed joint (11).
- ✓ Unscrew the screws (4) and lift support ring (5) slightly. First unscrew the stud bolt including the cable leading through it, then take off the support ring. Remove the remaining stud bolts (6).
- ✓ Pull the cup (7) and the threaded flange (12) up to remove it, turning it left and right. The cup has been screwed into the threaded flange, refer to detail Z.
- Remove the screws (14) and take off the pressure flange (15) and the pressure plate (16).
- Clean the cup and the pressure plate with water and dry these parts.
- ✓ Pull off the probe head (8) and pull the old electrolyte hose kit out of the probe head using a set of pointed pliers (refer to detail Y).

- Remove venting plug, clean the opening in the probe head and the venting plug with water and dry all parts.
- Unscrew the diaphragm bolt (29) of probe type 04 using the appropriate assembly tool. Clean the diaphragm area and the diaphragm bolt with water and dry these parts.

Probe	Part no.
diameter	of assembly tool
38 mm	591 293-
83 mm	591 294-

- Check the integrity of the diaphragm plug and probe ground surfaces.
- Replace the diaphragm bolt if damaged. If a damage is detected on the probe tube, the probe must be returned to the factory for repair.
- ✓ Screw in the diaphragm bolt manually and tighten it using a dynamometric key (part no. K01338-). Pass the point with the clicking sound two or three times.

Probe	Tightening	
diameter	torque	
38 mm	10 Nm	
83 mm	27 Nm	

- ▼ Only the outside of the diaphragm area of probe type 03 can be cleaned because the glass puck (28) is shrunkfit.
- Clean the electrolyte tube (23) at the upper end of the probe head with water and dry these parts.
- Check O-rings (24) and spacing ring (25) in the probe head for proper fit and insert the spacer (26) in the probe head, refer to detail Y below.
- ▼ Slightly lubricate the electrolyte tube with silicone grease or vaseline, place the probe head carefully on the electrolyte tube and turn it slightly while pushing it onto the O-rings. Turn the probe head until the venting plug has reached its proper position (refer to view A).
- ✓ Cut the new electrolyte hose kit to proper length (supplied in excessive lengths). Use the old electrolyte hose kit as a guide to determine the length. Cut the PTFE hose with a sharp knife at an angle (refer to detail X) on both sides and make sure not to damage the thread (10). Cut the thread until it protrudes 4-5 cm from the end of the hose. It is important to cut the hose at an angle to ensure that the electrolyte can flow out of the hose easily.
- ✓ Lubricate the gasket elements (17)-(20) on the new electrolyte hose with silicone grease or vaseline.
- ▼ Wipe the PTFE hose clean and push it fully into the probe through the opening at the probe head. If the hose is too long, it must be shortened. Otherwise, it might get bent at the slanted end and prevent or obstruct electrolyte flow (refer to detail X). Observe the proper fit of the gasket parts in the probe head (refer to detail Y, above).
- Screw venting plug into the probe head.
- Place the pressure plate and the pressure flange on the probe head, install the screws by tightening crosswise.

- ▼ Slightly lubricate the O-ring (13) with silicone grease or vaseline and place the cup with the threaded flange on the pressure plate.
- Screw the stud bolt manually into the pressure flange and tighten it.
 Important: The stud bolt with the cable must be turned into the hole opposite to the venting plug.
- Place the support ring in the stud bolts, turn the screws into the stud bolts and tighten screws crosswise.
- Insert cable into connection housing and connect it to terminal 2. Carefully screw down the cover.
- Unscrew the reference electrode from the T piece and fill the reservoir with electrolyte. Screw the reference electrode back in and place the connector on the reference electrode.
- Connect the air hose and apply pressure to the electrolyte reservoir.
- ▼ Open the venting plug on the probe head. Close the venting plug again as soon as the electrolyte emerges without bubbles. The probe has now been vented and is fully operative again. For venting, the pressure applied should be at least 2 to 3 bar. Prior to venting, please make sure that there is enough electrolyte in the reservoir. The electrolyte emerging while venting can be collected an reused, provided that it is collected in a clean reservoir.

11.10 Assembly instructions for the probe head of the ring probe

Due to the structural features and the very short electrolyte line, no electrolyte hose kit is available for the ring probe, in contrast to the rod probes. Clogging of the electrolyte line occurs very seldom with this probe and can normally be corrected by simply flushing the line with water and compressed air. If it is necessary to remove the probe head, you should proceed as follows.

Removal and assembly instructions (for item numbers refer to Figure 24)

- Fully unscrew out the 4 low-lying pan head screws (8) at the probe head (9).
- ▼ Pull the probe head out of the probe body by turning it slightly.
- Fully turn out the straight male union of the electrolyte supply line (1).
- ▼ Pull the reducing fitting (3) and the PTFE hose (5) out of the probe head and clean these parts. Replace any damaged parts.



Fig. 24 Sectional drawing of the probe head

- Clean the diaphragm area inside and outside with water and dry these parts.
- ▼ Reinstall the reducing fitting and the PTFE hose by proceeding as follows:
- Pull thread (4) in reducing fitting and secure it with O-ring (3). Lead thread from opening "C" to opening "D". Push reducing fitting fully into opening "C". The thread should now show in opening "D". Pass the thread through the PTFE hose starting at the straight end.
- ▼ Push PTFE hose with the thread stretched fully into opening "D". Secure the thread at the slanted side of the PTFE hose with a hose ring.
- ▼ Fill the O ring grooves on the probe head with silicone grease and push on the O rings (7).
- ✓ Push the probe head into the glassed opening of the ring probe and fix it with the 4 pan head screws (tighten crosswise).

11.11 Troubleshooting

Table 7 Troubleshooting checklist

Malfunction	Cause	Remedy
Display oscillates	Insufficiently vented	Venting
Display oscillates when touching the	a Insufficiently vented	Venting
electrolyte hose	b High-impedance diaphragm	Increase pressure or clean diaphragm
	c Reference electrode exhausted	Replace reference electrode
Display oscillates when mixing	a Loose connection	Check wiring
	b Unfavorable place of installation	Choose other place of installation, e.g., behind a baffle
	c High-impedance diaphragm	Clean diaphragm
	d Pt 100 defective	Install new Pt 100
Display goes to 0 or full scale	a Reference electrode defective or element exhausted, for test refer to 11.4. Check connector or contact pin of reference electrode or cable	Replace reference electrode
	 b Isolation amplifier (IA) defective Measure output voltage with multimeter, short-out IA input or apply a voltage of max. 1 Volt using the simulator IA must transmit input voltage in 1:1 ratio, otherwise it is defective 	Replace isolation amplifier
	c Resistor of Pt 100 defective	Check resistor. Must be replaced by Pfaudler technician if defective
	 d If no fault under a, b or c, perform calibration in insulated reservoir Read voltage with grounded and non-grounded buffer solution. Max. deviation of ±1 mV or ±0.1 pH, ground pH probe! 	Repair by Pfaudler if cable and IA are ok
Sudden change of pH display without change in pH	Electrolyte hose too old	Replace electrolyte hose
Display oscillates or full-scale deflection when touching the probe tube or	a Air in electrolyte line	Vent probe
cable, or display oscillates or full-scale deflection during calibration	b Derivative conductor inside the glass ruptured	Repair by Pfaudler
Identical display with different buffer solutions	 Pore in pH glass (product is in contact with derivative layer), potential approx400 mV in different buffers 	Repair by Pfaudler
	b Isolation amplifier defective, no power supply	Check and replace
	c Cable or transmitter input defective	Replacement
Zero drift	a Reference electrode exhausted or defective	Replace reference electrode
Zero point no longer in permitted range Zero drifts when venting the probe	b Defective cable or insulation fault caused by moisture	Check cable and probe terminal box, remove moisture with hot air blower
	c Power supply of isolation amplifier was disconnected for a long time	Repair by Pfaudler
	d Electrolyte hose too old?	Replace electrolyte hose
Slope too low or very slow response	a Determine potential at pH 3 and pH 7 Slope ≥ 55 mV/pH at 25 °C Slope too small	Lime deposit possible? Keep probe for 30 min in 10% HCl solut- ion, rinse in water and measure again
	b If acid treatment is not successful	Repair by Pfaudler
	c Insulation fault due to moisture?	Check cable and probe terminal box, remove moisture with hot air blower

11.12. Spare parts list

Spare parts for rod probe type 03/04	Part no.
pH pressure flange, complete (only for type N)	591 362-
– Straight male union with 0 ring DN 5/8, G 1/4", made of PVDF, natural	025 142-
pH probe head, complete	591 270-
– Venting plug on probe head M24 made of PVDF, white	591 122-
– O-ring for venting plug 12 x 3 mm made of Viton A	024 163D
Terminal box (< DN100) made of aluminum	591 014-
Terminal box (> DN150) made of aluminum	591 866-
– Gasket for terminal box made of rubber	591 015D
– Connection adapter, complete	595 610-
– Cover for terminal box made of aluminum	029 442-
– Gasket for cover made of rubber	591 082D
Explosion-proof ground terminal M5 Electrolyte hose kit for probe length 300 mm Electrolyte hose kit for probe length 450-950 mm Electrolyte hose kit for probe length 1150-2150 mm Electrolyte hose kit for probe length 2500-3250 mm Diaphragm bolt for probe diameter 38 mm Diaphragm bolt for probe diameter 83 mm Assembly tool for diaphragm bolt for probe diameter 83 mm Dynamometric key, square, 3/8", 10-60 Nm Protective cap, complete, for probe diameter 38 mm, made of PE black Protective cap, complete, for probe diameter 83 mm, made of PE black Telescopic rod, length 1.5 - 4.5 m Angular cup for telescopic rod, capacity of 2 l	029 422- 591 8931 591 8932 591 8933 591 8934 591 138E 591 130E 591 293- 591 294- K01 338- 591 076- 591 075- K07 055- K07 054-
Spare parts for ring probe	Part no.
Probe head pH ring, made of PVDF, white, without built-in parts	591 809-
– O-ring for probe head 14 x 1.5 mm made of Viton A	063 904-
Venting plug on probe head PG7 made of PVDF, white	591 810-
– O-ring for venting plug 8 x 1.6 mm made of Viton A	024 128D
Straight male union with O ring DN 5/8, G 1/4", made of PVDF, natural	025 142-
Angular connector for hoses, 3 mm	029 227D
Terminal box made of aluminum	591 953-
– Gasket for terminal box made of rubber	591 909D
Connection adapter, complete, for pH/rH/LF probes	595 610-
Explosion-proof ground terminal M5	029 422-
Resistance thermometer Pt100, diameter 3.2 mm	591 343-
Blanking plug PG7	029 015D

Spare parts for electrolyte reservoir type K	Part no.
Electrolyte reservoir type K, 0.7 liters	591 748-
Connector and cable for reference electrode	591 368-
Plug with rim, diameter 10 mm, made of silicone	035 246D
T connection, complete	595 711-
Cup 0.7 liters made of PC, transparent	591 788-
Pressure plate made of PA6	591 747-
– O-ring for pressure plate 50 x 3 mm made of Viton A	024 149-
Pressure flange	024 150-
Threaded flange	591 749-
Support ring made of PA6	591 751-
Stud bolt M10 x 215 mm made of 1.4571	591 753-
Protective tube M10 x 280 mm made of 1.4571	591 752-
Cable gland PG7 made of PA, gray	029 352D
Spare parts for electrolyte reservoir type N	Part no.
Electrolyte reservoir type N, 1.7 liters, without isolation amplifier	255 294-
Electrolyte reservoir type N, 1.7 liters, with one isolation amplifier	255 2941
Electrolyte reservoir type N, 1.7 liters, with two isolation amplifiers	255 2942
Connector and cable for reference electrode	591 368-
Venting plug M10, length 45 mm, made of POM, black	591 885-
– O-ring for venting plug 9 x 2 mm made of Viton A	024 268D
Shut-off valve with non-return valve, G 1/4", M8, made of PVDF, white	595 719-
– O-ring for shut-off valve 6 x 3 mm made of Viton A	024 381D
Cup P4-25 made of PC, transparent yellow	029 133-
Connection cover	591 891-
– O-ring for connection cover 120 x 4 mm made of Viton A	024 123-
Ball valve, complete, DN 4, G 1/4", made of PVDF, natural	029 137-
– Connection nipple G 1/4" made of PVDF, white	254 952-
– O-ring for connection nipple 12 x 2 mm made of Silicone	029 729-
– O-ring 10 x 2 mm made of Viton A	024 105D
– PTFE disk	595 870-
– Straight male union with O ring DN 5/8, G 1/4", made of PVDF, natural	025 142-
Hose 6 x 1 mm, length 10 m, made of PA11, transparent	029 359D
Mounting plate for isolation amplifier	591 887-
– Screws for fixing the mounting plate (2 pce), M8x16	031 102D
$\label{eq:main_series} \begin{array}{l} \mbox{Miscellaneous} \\ \mbox{Reference electrode type AgAgCl} \\ \mbox{Reference electrode type AgAGel} \\ \mbox{Electrolyte reservoir KCl, 3 x 1 liter} \\ \mbox{Electrolyte reservoir K_2SO_4, 3 x 1 liter} \\ \mbox{Pfaudler connection cord with special connector, 3 m} \\ \mbox{Pfaudler connection cord with special connector, 5 m} \\ \mbox{Pfaudler connection cord with special connector, 10 m} \\ \mbox{Isolation amplifier type 82 G} \\ \mbox{Power supply unit type WG 10 A7} \\ \mbox{Integrator 0.33 } \mu \\ \mbox{F} \end{array}$	Part no. 591 669- 595 860- 591 383- 591 081- 254 6343 254 6343 254 6345 254 6340 068 607- 029 415- 068 262-

L <u>b</u>	Physikalisch-Technische Bundesanstalt Braunschweig und Berlin	Physikalisch-Technische Bundesanstalt Braunschweig und Berlin
		(13) SCHEDULE
	EC TVPE EVAMINATION CEDTIEICATE	(14) EC-TYPE-EXAMINATION CERTIFICATE PTB 03 ATEX 2207 X
(1)		(15) Description of equipment
(2)		The measuring probes pH, types 03 04 03 dual 04 dual and 40 differential th types 03 and PH. are used to measure the pH-value and the readox notanizal not finuities in anomalice transforms or inhinous in the bazardous area
(9)) EC-type-examination Certificate Number: PTB 03 ATEX 2207 X	
(4)) Equipment: Measuring probes pH, types 03, 04, 03 dual, 04 dual and 40 differential, rh types A and B and pH/ rH types 03 and 04	Calegory 1/2-equipment The terminal housing is installed in hazardous areas requiring equipment of category 2. The
(5)	Manufacturer:	process connectors are mounted into the partition separating areas from each other where equipment of catagory 2 or 1 is required. The sense or is installed in the hazardous area for colonov 4 convincent for a participant of a catagory 10-barninement the moreas measure and the
(o) (E)	This equipment and any acceptable the documents therein referred to.	process temporatives to temporative and subject in the process temporative to the process temporative and the transpectively. In case of a deviation from these operating conditions it is hall be considered that the process does not how any estimation to be added to the process the matching the process of a deviation from the process of the process
(8)	The Physikalisch-Technische Bundesanstalt, notified body No. 0102 in accordance with Article 9 of the Council Directive 94/9/EC of 23 March 1994, certifies that this equipment has been found to comply with the Essential Health and Safety Requirements relating to the design and construction of equipment and protective systems intended for use in potentially explosive atmospheres, given in Annex II to the Directive.	to consume of the server does not show any service and the system as regards the pressures / temperatures of the media used. Marking: () II 2 G EEx ia IIC T6
	The examination and test results are recorded in the confidential report PTB Ex 04-20191.	Category 2-equipment
(6)) Compliance with the Essential Health and Safety Requirements has been assured by compliance with: EN 50014:1997 + A1 + A2 EN 50020:2002 EN 50284:1999	The measuring probes are installed in hazardous areas requiring category 2-equipment. For permissible operating temperatures and pressures reference is made to the manufacturer's
(10)	0) If the sign "x" is placed after the certificate number, it indicates that the equipment is subject to special conditions for safe use specified in the schedule to this certificate.	specifications. Electrical data
(11)		Marking: (EX) II 2 G Supply and signal circuit type of protection Intrinsic Safety EEx ia IIC only for connection to a certified intrinsically safe circuit
(12)	z) The marking of the equipment shall include the lonowing. (E) 11/2 G EEX ia IIB T6 or 112 G EEX ia IIC T6	L, negligibly low C, neglicibly low
	Zertifizierungsstelle Examistifiserunz By order:	Marking: (EX) II 1/2 G Warking: (EX) II 1/2 G type of protection Intrinsic Safety EEx ia IIB only for connection to accentified intrinsically safe circuit
	DrIng. U. Johannskerder S.	
I	EC-type-examination. Certificates without signature and official stamp shall not be valid. The certificates may be circulated	EC-type-examination Certificates without signature and official stamp shall not be valid. The certificates may be circulated only without alteration. Extratass or alterations are subject to approved by the Physicatisch-Technische Bundesentait.
	uny wuldur are atori. Extrato di area da aspecta per opprovany to approvany territo and	In case of dispute, the German text shall prevail. Physikalisch-Technische Bundeaanstalt • Bundesaliee 100 • 0.38116 Braunschweig

Types: pH 0 rH A, subject to the conditions and limits sp instructions.	suring probes 3, pH 04, 03 Dual, 04 Dual, 40 Differencial, , rH B, pH/rH 03, pH/rH 04
Types: pH 0 rH A, subject to the conditions and limits sp instructions.	3, pH 04, 03 Dual, 04 Dual, 40 Differencial, , rH B, pH/rH 03, pH/rH 04
instructions.	
	pecified in the technical documentation and the operating
potentially explosive atmospheres and stipulated in Annex II of Directive 94/9	measuring probes are suitable for their intended use in d comply with the essential safety and health requirement 9/EC. Other assembled equipment and components are clarations of conformity and their operating instructions.
	ype examination pursuant to Annex III PTB 03 ATEX 2207 X
Phys	ied body no. 0102 ikalisch-Technische Bundesanstalt (PTB) lesallee 100, D-38116 Braunschweig
	uction quality assurance pursuant to Annex IV EX2 03 05 45670 001
ΤÜV	ied body no. 0123 Product Service GmbH ieb-Daimler-Straße 7, D-70794 Filderstadt
Type of protection: 🔬 II	1/2G EEx ia IIB T6 bzw. II 2G EEx ia IIC T6
applied: EN 5	50014:1997+A1+A2 50020:2002 50284:1999
Community Directives applied: Explo	osion protection Directive 94/9/EC

capacitors in the supply circuits is 180 nF.

A3.1 Potentially hazardous atmospheres

The application of the pH measuring probe in potentially hazardous atmospheres belonging to zone 0 is permitted if the following conditions are maintained:

Required type of protection for category 1/2 equipment	EEx ia IIB
Power and signal circuits	For connection to certified, intrinsically safe electrical circuit only. The following maximum values apply to the measuring circuits of probes on a common probe carrier: $U_i = 30V$ $\Sigma I_i = 100 \text{ mA}$ $L_i \text{ (probe) negligible}$ $C_i \text{ (probe) negligible}$ $C_{connection cord} \le 10 \text{ nF}$
	The maximum allowable inductance of all inductors in the supply circuits is 11 mH. The maximum allowable capacitance of all

A3.2 Atmospheric conditions

The process pressure of the media or the media temperature must be in between 0.8 ... 1.1 bar or -20 ... 60 °C for all applications that require category 1/2 equipment. If these conditions of use are not complied with by the measuring sensor, it must be ensured that the measuring sensor does not heat up itself (not even in the event of fault). Please note that the safe operation of the plant with respect to pressures/temperatures of the substances used are the responsibility of the operator. The characteristic data supplied by the manufacturer shall be observed.

A3.3 Lightning protection

If the pH probe is installed in systems that must be protected against ignition hazards caused by strikes of lightning, the pH probe must be included in lightning protection. The lightning protection system must comply with the requirements of VDE 0165.

A3.4 Equipotential bonding

Since the intrinsically safe circuit of the measuring electrode is grounded during operation, a joint equipotential bonding system must exist in the course of the entire wiring of the intrinsically safe circuit and the reactor or pipeline must be included in this equipotential bonding system.

The information provided in this documentation corresponds to the state of the art at the time of printing. It is published in good faith. However, we will accept no warranty claims based on the information provided in this documentation. We reserve the right to include improvements, amendments and new findings in this documentation without prior notice. The actual design of products may deviate from the information contained in the calatoge if technical alterations and product improvements so require. The proposal made by Pfaudler for a concrete application will be binding in such cases.

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