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# Operating Instructions Liquistation CSF48

Automatic sampler for liquid media Calibration





# About this manual

This manual describes how to calibrate the distribution arm and the sample volume. In addition, it also explains all the possible ways of calibrating and adjusting digital sensors with the Memosens protocol.

### This manual does not include the following:

- Setup/General settings
  - --> Operating Instructions BA00443C "Commissioning"
- Display/Operation
  - --> Operating Instructions BA00443C "Commissioning"
- Inputs

   -> Operating Instructions BA00464C "Operation & settings"
- Outputs
   --> Operating Instructions BA00464C "Operation & settings"
- Sampling programs
   --> Operating Instructions BA00464C "Operation & settings"
- Additional functions
  - --> Operating Instructions BA00464C "Operation & settings"
- Data management
  - --> Operating Instructions BA00464C "Operation & settings"
- Expert
  - --> Internal Service Manual
- Diagnostics
  - --> Operating Instructions BA00463C "Maintenance & diagnostics"

# Safety instructions

# **A** CAUTION

### **The cleaning system is not switched off during calibration or maintenance activities** Risk of injury due to medium or cleaning agent

- ▶ If a cleaning system is connected, switch it off before removing a sensor from the medium.
- If you are not switching off the cleaning system because you wish to test the cleaning function, wear protective clothing, goggles and gloves or take other appropriate measures.

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# 1 Calibrating the distribution arm

It is only possible to calibrate the distribution arm in the version with multiple bottles.

The distribution arm must be calibrated if:

- The distribution arm motor has been replaced
- Error message "F236 Distribution arm" appears on the display

1. Select the number of bottles in the "Setup/Basic setup" menu.

2. Proceed as follows to calibrate the distribution arm:

#### Path: Menu/Calibration active

| Function   | Options | Info  |
|--|---------|---|
| Distribution arm   |         |   |
| ⊳Go to ref. point  | Action  | The reference run is started. The reference point is<br>in the middle at the front.<br>For versions with distributor plate, the reference<br>point is at the arrow in the middle of the plate. For<br>versions with distribution assembly the reference<br>point is between bottle number 1 and the last one. |
| With Adjust you can correct the distribution arm if the unit does not move to the reference point correctly. Use the two arrow keys to correct the position. |         |   |

3. Afterwards perform a distribution arm test in the "Diagnostics/Systemtest/Reset/Distribution arm" menu.

# 2 Calibrating the sample volume

### 2.1 Vacuum pump

The desired sample volume is set by manually adjusting the dosing tube.



Fig. 1: Vacuum pump

- 1 Outlet hose
- 2 Dosing chamber
- 3 Dosing chamber cover
- 4 Air hose connection
- 5 Lock for intake hose
- 6 Thread adapter nut for intake hose

Proceed as follows to calibrate the sample volume:

- 1. Check the sample volume set under Menu/Setup/General settings/Sampling/Dosing volume.
- 2. Release the thread adapter nut on the intake hose (item 6).
- 3. Turn the intake hose to the "open" position at the hose lock (item 5) and remove the hose from above.
- 4. Release the air hose (item 4) and remove the dosing chamber (item 2) from the front along with the outlet hose (item 1).
- 5. Open the bayonet lock (item 3) and open the dosing chamber.



- Dosing tube
- Allen screw
- Air hose connection

Fig. 2: Vacuum pump

- 1. Release the 2mm Allen screw with the key provided.
- 2. Set the sample volume by adjusting the dosing tube. Secure the dosing tube with the screw.Ise the white scale (A) to dose without pressure.

Use the blue scale (B) to dose with pressure.

- 3. Reinstall the parts in reverse order. Make sure that the contacts of the conductivity sensors are in the correct position.
- 4. Check that the dosing tube is set correctly by triggering a manual sampling routine.

### 2.1.1 Capacitance sample sensor

(Only for version with vacuum pump)

The capacitance sample sensor has been factory-adjusted, therefore, when first commissioning no adjustment is necessary. Adjust the sensor if the trigger sensitivity is too low. This is the case if the sample covers more than 30% of the sensor (item 1). The yellow and green lights (item 2) at the capacitance sample sensor are on.

Adjust the capacitance sample sensor as follows:

- 1. Make sure the dosing chamber is empty.
- 2. With the supplied screwdriver turn the adjusting screw (item 3) to the right (clockwise) until the yellow light goes out.
- 3. Then turn slightly back to the left (counterclockwise) until the yellow light is on again. At this point the sensor is adjusted to the most sensitive setting.
- 4. In order to verify the settings perform a manual sampling.
- 5. If the setting is too sensitive (causing e.g. false triggering, or the yellow light remains dark after sampling), adjust the sensor to a more insensitive setting by turning the adjusting screw further to the left.



Fig. 3: Adjust the capacitance sample sensor

- 1 Sensor
- 2 Yellow and green light
- 3 Adjusting screw

### 2.2 Peristaltic pump

In order to calibrate the sample volume, a measurement beaker with a volume of at least 200 ml is required.

Proceed as follows to calibrate:

#### Path: Menu/Calibration active

| Function  | Options                                      | Info   |  |  |
|---|--|--|--|--|
| Sample volume   | ▶ Sample volume                              |  |  |  |
| ▶ 1-point calibration   |  |  |  |  |
| Distributor position  | Options<br>- Front<br>- Bottle x<br>- Back   | Select the distributor position.                     |  |  |
| Sample volume   | 20 to 2000 ml                                | Set the sample volume.                               |  |  |
|   | Factory setting<br>100 ml                    |  |  |  |
| ▶ Start sampling  | Action                                       | The progress of the sampling operation is displayed. |  |  |
| Check whether the sample<br>Use ▷ Yes to repeat the sam   | volume is correct. Use ▶ No to er<br>npling. | iter the sample volume actually taken, e.g. 110 ml.  |  |  |
| ▶ 2-point calibration   |  |  |  |  |
| Use 2-point calibration for levels that fluctuate greatly.<br>The second sampling point must be either higher or lower (height difference of at least 1 m). |  |  |  |  |
| Distributor position  | Options<br>- Front<br>- Bottle x<br>- Back   | Select the distributor position.                     |  |  |
| Sample volume   | 20 to 2000 ml                                | Set the sample volume.                               |  |  |
|   | Factory setting<br>100 ml                    |  |  |  |
| Start 1. sampling   | Action                                       | The progress of the sampling operation is displayed. |  |  |
| Check whether the sample volume is correct. Use ► No to enter the sample volume actually taken, e.g. 110 ml. Use ▷ Yes to repeat the sampling.              |  |  |  |  |
| Start 2. sampling   | Action                                       | The progress of the sampling operation is displayed. |  |  |
| Check whether the sample volume is correct. Use > No to enter the sample volume actually taken, e.g. 110 ml. Use > Yes to repeat the sampling.              |  |  |  |  |

# 3 Calibration and adjustment

# 3.1 Definitions

### Calibration (as per DIN 1319):

A calibration is defined as a set of operations that establish the relationship between the measured value or expected value of the output variable and the related true or correct value of the measured variable (input variable) for a measuring system under specified conditions. A calibration does not alter the performance of the measuring device.

### Adjustment

An adjustment corrects the value displayed by a measuring device, in other words the measured/displayed value (the actual value) is corrected so that the reading agrees with the correct, set value.

The value determined during calibration is used to calculate the correct measured value and saved in the sensor.

# 3.2 Terminology

### 3.2.1 Zero point and slope

Using a mathematical function, the transmitter converts the input signal of the sensor y (raw measured value) to the measured value x. In many cases, this function is a simple linear of the form  $y = a + b \cdot x$ .

The linear element "a" is usually equivalent to the zero point and the factor "b" is the slope of the line.



Fig. 4: Linear function

a Zero point

b Slope

The **Nernst equation**, which is used to calculate the pH value, is a typical linear relationship:

 $\begin{array}{l} U_i = U_0 - \frac{2.303 \text{ RT}}{F} \text{ pH} \\ pH = -lg(a_{H^+}), a_{H^+} \dots \text{ activity of the hydrogen ions} \\ U_i \dots \text{ raw measured value in mV} \\ U_0 \dots \text{ zero point (=voltage at pH 7)} \\ R \dots \text{ relative gas constant (8.3143 J/molK)} \\ T \dots \text{ temperature [K]} \\ F \dots \text{ Faraday constant (26.803 Ah)} \end{array}$ 

The slope of the Nernst equation (-2.303RT/F) is known as the **Nernst factor** and has the value -59.16 mV/pH at 25 °C.

### 3.2.2 Delta slope

The device determines the difference in the slope between the calibration currently valid and the last calibration. Depending on the sensor type, this difference is an indicator of the condition of the sensor. The smaller the slope, the less sensitive the measurement, and the accuracy deteriorates particularly in the low measuring range.

Depending on the operating conditions, users can define limit values that represent the still tolerable absolute values of the slope and/or slope differentials. If the limit values are exceeded, maintenance must be performed on the sensor at the very least. The sensor must be replaced if the insensitivity problems persist after maintenance has been carried out.



Fig. 5: Delta slope

BlueLast calibrationRedCalibration currently valid∆bDelta slope

### 3.2.3 Delta zero point

The device determines the difference between the zero points or operating points (ISFET sensor) of the last and second-last calibration. A shift in the zero point or operating point (offset) does not alter the sensitivity of the measurement. However, if the offset is not corrected this can falsify the measured value.

As with the slope, you can also define and monitor limit values for the offset. If the limit values are exceeded, this means that maintenance must be performed on the sensor. For example, you may have to eliminate a blockage in the reference for the pH sensor.



Fig. 6: Delta zero point or operating point (ISFET sensor)

- a1 Zero point (operating point) of the second-last calibration
- a2 Zero point (operating point) of the last calibration
- ∆a Delta zero point (operating point)

# 3.3 Notes on performing a calibration

The following rules apply for all parameters:

- Calibrate in a way that reflects conditions in the process.
  - If the process medium is constantly moving, also move the calibration solution accordingly (e.g. use a magnetic stirrer if calibrating in the laboratory).
  - If your medium is relatively stationary, calibrate in solutions that are also stationary.
- Make sure that the samples are homogeneous for reference measurements, sample calibration etc.
- Avoid changes in the medium samples resulting from ongoing biological activity. Example: Use outlet water instead of a sample from the aeration basin for nitrate calibration.
- Use the same menu settings as those in the process to perform the calibration. Example: If you automatically compensate for the temperature effect during pH measurement, switch on automatic temperature compensation for the calibration also.
- It is advisable to perform the laboratory calibration using the "Memobase" database software (--> "Accessories"). This improves the availability of your measuring points and all the calibration and sensor data records are stored securely in the database.

# 4 pH sensors

# 4.1 Calibration intervals

### 4.1.1 Specifying the intervals

The service life of a pH glass electrode is limited. This is due, in part, to the deterioration and aging of the pH-sensitive membrane glass. This aging causes the gel-like layer to change and become thicker over time.

Symptoms of aging include:

- Higher membrane resistance
- Slow response
- Decrease in the slope

A change in the reference system (e.g. due to contamination, i.e. unwanted redox reactions at the reference electrode) or electrolyte solution dissolving away in the reference half cell can change the reference potential, which, in turn, causes a zero point shift in the measuring electrode.

To ensure a high level of accuracy, it is important to readjust the pH sensors at set intervals. The calibration interval depends heavily on the area of application of the sensor, as well as the required level of accuracy and reproducibility. The calibration interval can vary between daily and once every few months.

### Defining the calibration interval for the process

- 1. Check the sensor with a buffer solution, e.g. pH 7.
  - Proceed as specified in Step 2 only if the value deviates from the set point. No calibration/adjustment is necessary if the value is within the defined deviation tolerance range (see the Technical Information for the sensor).
- 2. Calibrate and adjust the sensor.
- 3. After 24 hours, check again with the buffer solution.
  - a. If the deviation is within the permitted tolerance range, increase the checking interval by doubling it for example.
  - b. If the deviation is larger, you must shorten the interval.
- 3. Continue to proceed as defined in Steps 2 and 3 until you have identified the suitable interval.

### Monitoring the calibration

- Define the limit values for monitoring the slope and zero point differentials (Menu/Setup/Inputs/pH/Extended setup/Diagnostics settings/Delta slope or Delta zero point).
  - └ These limit values depend on the process and must be determined by empirical means.

During calibration a diagnostics message is displayed if the defined warning limits have been exceeded. You then have to service the sensor by cleaning the sensor or reference, or by regenerating the glass membrane.

You have to replace the sensor if warning messages continue to be displayed despite the maintenance measure.

### 4.1.2 Monitoring the calibration interval

Once you have established the calibration intervals for your process, you can also have the device monitor them.

### Two functions are available to monitor the calibration interval:

- Calibration timer (Menu/Setup/Inputs/<Sensor type>/Extended setup/Calib. settings/Calibration timer)
  - You specify the calibration interval and the controller generates a diagnostics message once the set time has elapsed. You then recalibrate the sensor or replace it with a precalibrated sensor.

The timer is reset with the new calibration.

- Calibration validity (Menu/Setup/Inputs/<Sensor type>/Extended setup/Calib. settings/Calib. expiration date)
  - You set time limits to specify how long a calibration should be regarded as valid. Memosens sensors save all the calibration data. In this way it is easy to see whether the last calibration took place in the specified timeframe and is therefore still valid. This is particularly advantageous when working with precalibrated sensors.

# 4.2 Types of calibration

The following types of calibration are possible:

- Two-point calibration
  - With calibration buffers
  - Entry of data for the slope, zero point and temperature
- Single-point calibration
  - Entry of an offset or a reference value
  - Sample calibration with laboratory comparative value
- Temperature adjustment by entering a reference value

# 4.3 Two-point calibration

### 4.3.1 Applications and requirements

**Two-point calibration** is the preferred method for pH sensors, particularly in the following applications:

- Municipal and industrial wastewater
- Natural waters and drinking water
- Boiler feedwater and condensates
- Beverages

Calibrating with buffers with pH 7.0 and 4.0 is recommended for most applications. Alkaline buffer solutions have the disadvantage that carbon dioxide from the air can alter the pH value of the buffer on the long term. If calibrating with alkaline buffers it is best to do so in closed systems, such as flow assemblies or retractable assemblies with a rinse chamber, to minimize the effect of air.

### 4.3.2 With calibration buffers

You use calibration buffers to perform two-point calibration. The quality buffers supplied by Endress+Hauser are certified and measured in an accredited laboratory. The accreditation (DAR registration number "DKD-K-52701") confirms that the actual values and the maximum deviations are correct and traceable.

To calibrate the sensor, remove it from the medium and calibrate it in the laboratory. Since Memosens sensors save the data, you can always work with "precalibrated" sensors and do not have to stop monitoring the process to perform a calibration.

- 1. Go to the "CAL/2-pnt. calibration" menu.
- 2. Follow the instructions on the display.
- 3. Press "OK" **after** you have immersed the sensor into the first buffer.
  - └ The system starts calculating the measured value for the first buffer. Once the stability criterion is met, the measured value is displayed in mV.
- 4. Continue to follow the instructions.
- 5. Press "OK" after you have immersed the sensor into the second buffer.
  - The system starts calculating the measured value for the buffer. Once the stability criterion is met, the measured values of the two buffers and the calculated values for the slope and zero point are displayed.
- 6. Select "OK" when you are asked to accept the calibration data for adjustment.
- 7. Put the sensor back into the medium and press "OK" again.
  - └ This deactivates the hold and the system starts measuring again.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

Only use calibration buffers once.

### 4.3.3 Entering data for the zero point, slope and temperature

- You enter the slope, zero point and temperature manually. The function for determining the pH value is calculated from these values. Thus, the data entry returns the same result as two-point calibration.
- You must determine the slope, zero point and temperature in an alternative way.
- 1. Go to the "CAL/Data input" menu.
  - └ The slope, zero point and temperature are shown on the display.
- 2. Select each value one after another and then enter your desired numerical value.
  - └→ Since you are entering all the variables for the Nernst equation directly, no additional information is displayed by the controller.
- 3. Select "OK" when you are asked to accept the calibration data for adjustment.

You can cancel the calibration any time by pressing the "ESC" key. No new data are then used to adjust the sensor.

# 4.4 Single-point calibration

### 4.4.1 Applications and requirements

**Single-point calibration** is particularly useful if the deviation of the pH value from a reference value, and not the absolute pH value itself, is of interest to the user. Applications for single-point calibration include:

- Process control
- Quality assurance

Fluctuations in the process value should not exceed  $\pm 0.5$  pH and the process temperature must remain relatively constant. As the measuring range is limited as a result, it is possible to set the slope to -59 mV/pH (at 25 °C).

To adjust the sensor, you enter an offset or a reference value.

Alternatively, you can also use the "sample calibration". Here, you take a sample from the process and determine the pH value in the laboratory. In the case of the laboratory sample, you must make sure that the pH value is determined at the process temperature.

### 4.4.2 Sample calibration

- With this type of calibration, you take a sample of the medium and determine its pH value (at process temperature) in the laboratory. You then use this laboratory value to adjust the sensor. This does not change the slope of the calibration function.
- 1. Go to the "CAL/.../Sample calibration" menu.
- 2. Follow the instructions on the display.
- 3. Press "OK" after you have taken the sample.
  - └ The following message appears on the display:
  - Sample calibration.
- 4. Press the navigation button after you have determined the laboratory value.
  - ← A line appears where you can enter the laboratory value.
- 5. Enter your laboratory measured value here and then go to  $\triangleright$  Continue.
  - └ The measured value, laboratory value and the resulting offset (zero point for ISE) are displayed.
- 6. Accept the calibration data and then return to the measuring mode.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

### 4.4.3 Entering the offset or reference value

You either enter an offset or a (reference) measured value calculated beforehand. This then shifts the calibration function along the X-axis (pH). The slope is not affected.

1.Go to the "CAL/1-pnt. calibration" menu.

- 2. Decide which value you want to enter:
  - a. Offset
    - └ Enter the desired offset. Once accepted, the value you entered immediately affects the "Meas. value".
  - b. Meas. value
    - ← Enter the desired measured value. Once accepted, the value you entered immediately affects the "Offset".

3. Accept the calibration data and then return to the measuring mode.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

### 4.5 Temperature adjustment

- 1. Determine the temperature of your process medium with an alternative measurement, e.g. a precision thermometer.
- 2. Go to the "CAL/<Sensor type>/Temperature adjustment" menu.
- 3. Leave the sensor in the process medium and keep clicking "OK" until temperature measurement is started via the sensor.
- 4. Enter the reference temperature from the alternative measurement. You can either enter the absolute value or an offset for this purpose.
- 5. Once you have entered your data, click "OK" until the new data have been accepted.
  - └ This completes temperature sensor adjustment.

# 4.6 Error messages when performing the calibration

| Display message   | Causes and possible remedial measures  |
|---|--|
| The calibration is invalid. Do you want to start a new<br>calibration?<br>Slope out of tolerance.<br>Zeropoint out of tolerance.<br>Sample concentration too low. | <ul> <li>The calibration buffer is contaminated or the pH value is no longer within the permitted limits. As a result, the permitted measured value deviation is exceeded.</li> <li>Check the expiry date</li> <li>Use a fresh buffer</li> </ul>   |
|   | <ul> <li>Incorrect buffers used. As a result, the buffer recognition function - for example - does not work correctly.</li> <li>pH values of the buffers are too close together, e.g. pH 9 and 9.2</li> <li>Use buffers with a larger pH difference</li> </ul>   |
|   | Sensor aging or contaminated. As a result, the permitted limit<br>values for the slope and/or zero point are exceeded<br>Clean the sensor<br>Adjust the limit values<br>Regenerate or replace the sensor   |
| The stability criterion is not fulfilled. Do you want to repeat the last step?  | The measured value or temperature is not stable. As a result,<br>the stability criterion is not met.<br>• Keep the temperature constant during calibration<br>• Replace the buffer<br>• Sensor old or contaminated. Clean or regenerate.<br>• Adjust the stability criteria<br>(Menu/Setup/Inputs/ <electrode slot="">/Calib.<br/>settings/Stability criteria)</electrode> |
| Calibration aborted. Please clean sensor before<br>immersing in process medium. (Hold will be disabled)   | The user has aborted the calibration.  |

# 5 ORP sensors

# 5.1 Types of calibration

The following types of calibration are possible:

- Two-point calibration with samples of medium (only Main value = "%")
- Single-point calibration with calibration buffer
- Entry of data for an offset
- Temperature adjustment via reference value

# 5.2 Single-point calibration

### 5.2.1 General information

The buffers contain ORP pairs with a high exchange current density. Such buffers have the advantage of higher accuracy levels, better reproducibility and faster measurement response times.

Temperature compensation does not take place when measuring the ORP since the thermal behavior of the medium is not known. However, the temperature is indicated with the measurement result and for this reason it makes sense to adjust the temperature sensor at process-dependent intervals.

### 5.2.2 Single-point calibration with calibration buffers

- With this type of calibration, you work with calibration buffers, e.g. ORP buffers from Endress+Hauser. For this purpose, you remove the sensor from the medium and calibrate it in the laboratory. Since Memosens sensors save the data, you can always work with "precalibrated" sensors and do not have to stop monitoring the process for extended periods to perform a calibration.
- 1. Go to the "CAL/1-pnt. calibration" menu.
- 2. Follow the instructions on the display.
- 3. Accept the calibration data and then return to the measuring mode.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

#### 5.2.3 Entering data for an offset

- You enter the offset directly with this type of calibration. Use the measured value of a reference measurement, for example, to determine the offset.
- 1. Go to the "CAL/Data input" menu.
  - └ The current offset is displayed.
- 2. Decide whether you want to keep this value or enter a new value.
- 3. Enter the new offset.
- 4. Accept the calibration data and then return to the measuring mode.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

# 5.3 Two-point calibration (only ORP %)

- You must adapt the sensor to your process to obtain useful ORP % values. This is achieved through two-point calibration. The two calibration points are characteristic of the most important states your medium can assume in the process. You require two different compositions of your medium that represent the characteristic limits of your process (e.g. 20% and 80% value). The absolute value in mV is not relevant for the ORP % measurement.
- 1. Go to the "CAL/Redox/2-pnt. calibration" menu.
- 2. Follow the instructions on the display.
- 3. Accept the calibration data and then return to the measuring mode.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

### 5.4 Temperature adjustment

- 1. Determine the temperature of your process medium with an alternative measurement, e.g. a precision thermometer.
- 2. Go to the "CAL/<Sensor type>/Temperature adjustment" menu.
- 3. Leave the sensor in the process medium and keep clicking "OK" until temperature measurement is started via the sensor.
- 4. Enter the reference temperature from the alternative measurement. You can either enter the absolute value or an offset for this purpose.
- 5. Once you have entered your data, click "OK" until the new data have been accepted.
  - └ This completes temperature sensor adjustment.

# 5.5 Error messages when performing the calibration

| Display message   | Causes and possible remedial measures   |
|---|---|
| The calibration is invalid. Do you want to start a new calibration?                                     | <ul> <li>The calibration buffer is contaminated or the ORP potential is no longer within the permitted limits. As a result, the permitted measured value deviation is exceeded.</li> <li>Check the expiry date</li> <li>Use a fresh buffer</li> </ul>                                     |
| The stability criterion is not fulfilled. Do you want to repeat the last step?                          | <ul> <li>The measured value is not stable. As a result, the stability criterion is not met.</li> <li>Replace the buffer</li> <li>Sensor old or contaminated. Clean or regenerate.</li> <li>Adjust stability criteria (Menu/Setup/Inputs/pH/Calib. settings/Stability criteria)</li> </ul> |
| Calibration aborted. Please clean sensor before<br>immersing in process medium. (Hold will be disabled) | The user has aborted the calibration.   |

# 6 Conductivity sensors

# 6.1 Types of calibration

The following types of calibration are possible:

- Cell constant with calibration solution
- Installation factor (only inductive sensors)
- Air set (residual coupling, only inductive sensors)
- Temperature adjustment via reference value

# 6.2 Cell constant

### 6.2.1 General information

A conductivity measuring system is generally calibrated in such a way that the exact cell constant is determined or checked using suitable calibration solutions. This process is described in the standards EN 7888 and ASTM D 1125, for example, and the method for producing a number of calibration solutions is explained. Another alternative is to purchase international calibration standards from national measurement institutes. This is particularly important in the pharmaceutical industry, which requires a calibration to be traced to internationally recognized standards. To calibrate its testing units, Endress+Hauser uses special reference material (SRM) from the US National Institute of Standards and Technology (NIST).

### 6.2.2 Calibrating the cell constant

- You enter a reference value for the conductivity with this type of calibration. In addition, you specify how the system should compensate for the influence of temperature. In the result, the controller calculates a new cell constant for the sensor.
- 1. Go to the "CAL/Cell constant" menu.
- 2. Work your way through the menu functions that follow.
- 3. Start the calibration.

| Function            | Options                       | Info   |
|---------------------|-------------------------------|--|
| Current cell const. | Read only                     | Value currently saved in the sensor  |
| Temp. compensation  | Options<br>• No<br>• Yes      | As an alternative to the compensated conductivity (Yes)<br>you can also determine the cell constant by calibrating<br>the uncompensated conductivity (No). |
|                     | <b>Factory setting</b><br>Yes |  |

#### Path: CAL/Conductivity/Cell constant

| Function            | Options   | Info   |
|---------------------|---|--|
| Coeff. Alpha        | 0.00 to 20.00 %/K<br>Factory setting<br>Depends on the sensor                         | <i>Temp. compensation = "Yes"</i><br>The alpha coefficients and alpha reference temperatures<br>of Endress+Hauser can be found in the documentation<br>supplied with the calibration solutions.<br>Enter the appropriate values. |
| Alpha ref. temp.    | -5.0 to 100.0 °C<br>(23.0 to 212.0 °F)<br><b>Factory setting</b><br>25.0 °C (77.0 °F) |  |
| Temp. source        | Options<br>Sensor<br>Manual<br>Factory setting<br>Sensor                              | <ul><li>Decide how you want to compensate the medium temperature:</li><li>Automatically using the temperature sensor of your sensor</li><li>Manually by entering the medium temperature</li></ul>                                |
| Medium temperature  | -50.0 to 250.0 °C<br>(-58.0 to 482.0 °F)<br>Factory setting<br>25.0 °C (77 °F)        | <i>Temp. source = "Manual"</i><br>Enter the temperature of your medium.  |
| Conductivity ref.   | 0.000 to 2000000 μS/cm<br>Factory setting<br>0.000 μS/cm                              | Temp. compensation ="Yes"<br>Enter the compensated conductivity of your calibration<br>solution here.<br>Temp. compensation ="No"<br>Enter the uncompensated conductivity of your<br>calibration solution here.                  |
| ▷ Start calibration | Start the calibration. Follow the instructions on the display.                        |  |

#### Path: CAL/Conductivity/Cell constant

You can cancel the calibration any time by pressing the "ESC" key. No new data are then used to adjust the sensor.

# 6.3 Air set (residual coupling, only inductive sensors)

While the calibration line goes through zero for physical reasons in the case of conductive sensors (a current flow of 0 corresponds to a conductivity of 0), when working with inductive sensors, the residual coupling between the primary coil (transmitter coil) and secondary coil (receiver coil) must be taken into account or compensated for. The residual coupling is not only caused by the direct magnetic coupling of the coils but also by crosstalk in the supply cables. For this reason, the process of commissioning an inductive sensor always starts with an "air set". Here, the sensor is connected to the transmitter with the cables provided, held in air in a dry state (zero conductivity) and an air set calibration is performed at the transmitter. The cell constant is then determined using a precise calibration solution, as is the case with conductive sensors.

Sensors with a Memosens protocol are already calibrated at the factory and their residual coupling generally does not have to be adjusted on site.

# 6.4 Installation factor (only inductive sensors)

In confined installation conditions, the wall affects conductivity measurement in the liquid. The installation factor compensates for this effect. The transmitter corrects the cell constant by multiplying by the installation factor.

The size of the installation factor depends on the diameter and the conductivity of the pipe nozzle, as well as the distance between the sensor and the wall.

If there is a sufficient distance between the wall and the sensor (a > 15 mm (0.59"), from DN 80), the installation factor f does not have to be taken into consideration (f = 1.00). If distances from the wall are smaller, the installation factor is bigger for electrically insulating pipes (f > 1), and smaller for electrically conductive pipes (f < 1).

It can be measured using calibration solutions, or a close approximation determined from the

following diagram.



Fig. 7: Relation between the installation factor f and the wall distance

1 Electrically conductive pipe wall

2 Electrically insulating pipe wall

### 6.4.1 Calibrating the installation factor

- 1. Go to the "CAL/Cond i/Inst. factor/Calibration" menu.
- 2. Work your way through the menu functions that follow.

#### Path: CAL/Cond i/Inst. factor/Calibration

| Function            | Options  | Info  |
|---------------------|--|---|
| Cur. inst. factor   | Read only  | Value currently saved in the sensor   |
| Temp. compensation  | Options<br>• No<br>• Yes<br>Factory setting<br>Yes                             | As an alternative to the compensated conductivity (Yes)<br>you can also determine the cell constant by calibrating<br>the uncompensated conductivity (No).  |
| Coeff. Alpha        | 0.00 to 20.00 %/K  | Temp. compensation = "Yes"  |
|                     | <b>Factory setting</b><br>Depends on the sensor                                | The alpha coefficients and alpha reference temperatures<br>of Endress+Hauser can be found in the documentation  |
| Alpha ref. temp.    | -5.0 to 100.0 °C<br>(23.0 to 212.0 °F)   | Enter the appropriate values.   |
|                     | Factory setting<br>25.0 °C (77.0 °F)   |   |
| Temp. source        | Options<br>• Sensor<br>• Manual  | Decide how you want to compensate the medium<br>temperature:<br>• Automatically using the temperature sensor of you   |
|                     | <b>Factory setting</b><br>Sensor   | <ul> <li>Sensor</li> <li>Manually by entering the medium temperature</li> </ul>   |
| Medium temperature  | -50.0 to 250.0 °C<br>(-58.0 to 482.0 °F)<br>Factory setting<br>25.0 °C (77 °F) | <i>Temp. source = "Manual"</i><br>Enter the temperature of your medium.   |
| Conductivity ref.   | 0.000 to 2000000 µS/cm<br>Factory setting<br>0.000 µS/cm                       | Temp. compensation ="Yes"<br>Enter the compensated conductivity of your calibration<br>solution here.<br>Temp. compensation ="No"<br>Enter the uncompensated conductivity of your<br>calibration solution here. |
| ▶ Start calibration | Start the calibration. Follow the instructions on the display.                 |   |

You can cancel the calibration any time by pressing the "ESC" key. No new data are then used to adjust the sensor.

### 6.4.2 Entering the installation factor

- 1. Go to the "CAL/Cond i/Inst. factor/Entry" menu.
  - └ The installation factor currently used is displayed.
- 2. New inst. factor: Enter the installation factor, which you took from  $\rightarrow$   $\square$  7for example.
- 3. Start the calibration.

You can cancel the calibration any time by pressing the "ESC" key. No new data are then used to adjust the sensor.

### 6.5 Temperature adjustment

- The temperature sensor has to be calibrated at regular intervals to ensure the measured value is not falsified by incorrect temperature measurement.
- 1. Go to the "CAL/Conductivity/Temperature adjustment" menu.
  - The offset (of the last calibration) and the actual temperature value are shown on the display.
- 2. Mode
  - └ Decide which mode to use for temperature adjustment
  - a. 1-point calibration
    - └ You measure the medium temperature with a reference measurement and use this value to adjust the temperature sensor.
  - b. 2-point calibration
    - └ You use two samples of different temperatures.
  - c. Table
    - Adjustment based on data entries. You enter value pairs comprising the measured temperature of the temperature sensor and the related reference temperature. The temperature function is calculated from these value pairs. Press "SAVE" once you have entered all the points and select "OK" to confirm you want to accept the calibration data.
- 4. Follow the instructions on the display.
- 5. Accept the calibration data and then return to the measuring mode.

You can cancel the calibration any time by pressing the "ESC" key. No new data are then used to adjust the sensor.

# 6.6 Error messages when performing the calibration

| Display message   | Causes and possible remedial measures   |
|---|---|
| The calibration is invalid. Do you want to start a new calibration?                                     | Calibration solution exhausted. As a result, the permitted<br>measured value deviation is exceeded<br>• Check the expiry date<br>• Use a fresh calibration solution |
| Currently no calibration possible due to sensor failure.  | Sensor communication problem<br>• Replace sensor<br>• Contact the Service Department  |
| Calibration aborted. Please clean sensor before<br>immersing in process medium. (Hold will be disabled) | The user has aborted the calibration.   |

# 7 Oxygen sensors

### 7.1 Signal generation with amperometric sensors

The amperometric oxygen sensor is based on the reduction of oxygen at the noble metal cathode of a system filled with electrolyte.

Oxygen coming from the medium (e.g. air) diffuses through a membrane into the electrolyte film and is reduced at the cathode.

This means that there is practically no molecular oxygen at the cathode. Intense oxygen consumption takes place here and the oxygen partial pressure approaches zero.

The oxygen partial pressure of the medium is present in front of the membrane. This pressure is approx. 209 hPa in water vapor-saturated air under reference conditions (1013 hPa, 20°C). The partial pressure acts as the driving force to transport oxygen molecules through the membrane. The membrane acts as a diffusion barrier, i.e. oxygen molecules permeate the membrane depending on the difference in partial pressure.

In summary, an amperometric oxygen sensor has two important features:

- 1. The rate of oxygen consumption at the cathode is extremely high. Oxygen permeates the membrane depending on the external oxygen partial pressure (the internal pressure is practically zero) the external oxygen partial pressure is the driving force.
- 2. Due to the diffusion-inhibiting properties of the membrane, the flow of oxygen through the membrane and thus the electric signal current subsequently generated is in direct proportion to the oxygen partial pressure in front of the membrane, i.e. the sensor provides a linear signal current that depends on the oxygen partial pressure.

The amperometric oxygen sensor is therefore an oxygen partial pressure sensor.

# 7.2 Calibration intervals

### 7.2.1 Specifying the intervals

The calibration intervals depend heavily on:

- The application
- The sensor installation position

If you want to calibrate the sensor intermittently for a special application and/or on account of a special type of installation, you can calculate the intervals using the following method:

Inspect the sensor one month after commissioning for example:

- 1. Remove the sensor from the medium.
- 2. Clean the outside of the sensor with a damp cloth.
- 3. Then dry the sensor diaphragm carefully with a paper towel for example. (only amperometric sensors)
- 4. After 20 minutes, measure the oxygen saturation index in air.
- 5. Protect the sensor against external influences such as sunlight and wind.
- 6. Decide whether to calibrate depending on the result:
  - a. Amperometric sensor:
    - → If the measured value is not  $102 \pm 2$  %SAT, you must calibrate the sensor.
  - b. Optical sensor:
    - → If the measured value is not  $100 \pm 2$  %SAT, you must calibrate the sensor.
  - c. Otherwise extend the period until the next inspection.
- 4. Proceed as explained in Point 1 after two, four or eight months to determine the optimum calibration interval for your sensor.

Amperometric sensors only: Make sure to calibrate the sensor at least once a year.

### 7.2.2 Monitoring the calibration interval

Once you have established the calibration intervals for your process, you can also have the device monitor them.

### Two functions are available to monitor the calibration interval:

- Calibration timer (Menu/Setup/Inputs/<Sensor type>/Extended setup/Calib. settings/Calibration timer)
  - You specify the calibration interval and the controller generates a diagnostics message once the set time has elapsed. You then recalibrate the sensor or replace it with a precalibrated sensor.

The timer is reset with the new calibration.

- Calibration validity (Menu/Setup/Inputs/<Sensor type>/Extended setup/Calib. settings/Calib. expiration date)
  - You set time limits to specify how long a calibration should be regarded as valid. Memosens sensors save all the calibration data. In this way it is easy to see whether the last calibration took place in the specified timeframe and is therefore still valid. This is particularly advantageous when working with precalibrated sensors.

# 7.3 Types of calibration

The following types of calibration are possible:

- Slope
  - Air, water vapor-saturated
  - Air-saturated water
  - Air, variable
  - Data entry
- Zero point
  - Single-point calibration in nitrogen or oxygen-free water
  - Data entry
- Sample calibration
  - Slope
  - Zero point
- Temperature adjustment

Furthermore, the calibration menu for amperometric sensors contains two additional functions to reset the sensor's internal counters:

- Change electrolyte
- Change sensorcap

# 7.4 Slope calibration

### 7.4.1 General principles

In the case of slope calibration, the dependency on partial pressure is used to compare the signal current to a known, available reference - air.

The composition of dry air is known:

- 20.95 % oxygen
- 79.05 % nitrogen and other gases

### Altitude and partial pressure

The oxygen partial pressure otherwise only depends on the altitude or the current absolute air pressure. At an air pressure of 1013 hPa at sea level, the oxygen partial pressure is approximately 212 hPa. The absolute pressure and thus also the oxygen partial pressure change depending on the altitude. Using the barometric formula, the expected oxygen partial pressure can be calculated with only marginal errors up to a height of several kilometers. As a result, calibration is independent of the altitude.

### Three methods for obtaining reliable values for the absolute pressure of air

- 1. Using the altitude and the barometric formula which provide the correlation between the expected value for the average absolute air pressure and the altitude (also saved and accessible in the transmitter or sensor).
- 2. By measuring the absolute pressure of air with a pressure cell, for example.
- 3. The relative air pressure reduced to sea level is often available from weather reports. This relative air pressure can be converted to the absolute value using the barometric formula.

#### Water vapor

In reality, water in the form of water vapor is also always present in air. This is a contributing factor to the total pressure. This means that the water vapor in the air changes the oxygen partial pressure.

However, air can only hold a specific maximum volume of water. The rest is given off as condensate in liquid form (e.g. drops). The maximum amount of water vapor in air depends on the temperature and follows known functions.

### Air 100% rh

In this calibration model, the percentage of water vapor is deducted on the basis of the altitude and temperature so that information is available on the oxygen partial pressure actually present. For this model to work correctly, the sensor to be calibrated must be close to a water surface or be located in the headspace of a vessel partially filled with water. In this way, oxygen sensors can be precisely calibrated in a wide variety of applications, ranging from power stations to water treatment.

#### H2O air-saturated

After an adequate amount of time, water which has been aerated sufficiently is in equilibrium with the oxygen partial pressure of the air above the water. The "H2O air-saturated" calibration model uses this property. Here too, the model uses the temperature value to automatically reference back to the expected oxygen partial pressures. This model is often used to measure oxygen in closed tanks, such as fermenters filled with water.

### Air variable

This calibration model is for all applications in which the air pressure and air humidity in the vicinity of the sensor do not correspond to the standard atmospheric values previously mentioned, but are still known. Both variables can be specified here. The model is used, for example, for installed sensors that should be calibrated in operation at known conditions, such as in dry rinse air at 1020 hPa.

### Sample calibration

Sample calibration is another calibration option. Here, the measured value of the sensor is adjusted to a reference, obtained externally, of the same medium.

### 7.4.2 Calibration in the media mentioned

The calibration procedure is identical regardless of whether you are calibrating in water vapor-saturated air, air-saturated water or variable air:

- 1. Go to the "Oxygen/Oxygen/Slope" menu.
- 2. Choose between "Air 100% rh", "H2O air-saturated" and "Air variable".
- 3. Follow the instructions on the display.
- 4. Accept the calibration data and then return to the measuring mode.
- 5. Follow the instructions and then press "OK".

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

### 7.4.3 Data entry

- 1. Go to the "CAL/Oxygen/Slope/Data input" menu.
- 2. Select "New slope" and enter the new value.
- 3. Then select "OK" when you are asked to accept the calibration data for adjustment.
  - └ The new slope is used.

# 7.5 Calibration of the zero point

### 7.5.1 General principles

The zero point is not so important when working with relatively high concentrations of oxygen. This situation changes, however, as soon as oxygen sensors are used in the trace range and calibration should be performed in the zero point.

Zero point calibrations are demanding as the ambient medium - usually air - already has a high oxygen content. This oxygen must be excluded for the zero point calibration of the sensor and existing residual oxygen must be eliminated from the sensor environment.

There are two possible ways to do so:

- 1. Calibration of the zero point in a flow assembly that has been rinsed with gaseous nitrogen of acceptable quality (N5).
- 2. Calibration in zero solution. An aqueous solution of Na<sub>2</sub>SO<sub>3</sub> depletes oxidizing media and guarantees an oxygen-free environment after an adequate amount of time in airtight conditions.

### General rule for zero solutions

A solution of  $1g Na_2SO_3$  in 1 l water at approx.  $30^{\circ}C$  in a vessel that tapers towards the neck (e.g. Erlenmeyer flask or similar) is free from oxygen after approx. 0.5 h. When sealed airtight, this state is maintained for around 24 hours. This time is reduced if air is admitted.

### Prior to sensor zero point calibration

Has the sensor signal settled and is it steady?

Is the value displayed plausible?

If the oxygen sensor is calibrated too early, this can result in an incorrect zero point. As a general rule, operate the sensor in a zero solution for 0.5 hours and then assess the signal current in the steady state.

If the sensor was operated in the trace range before zero point calibration, the time specified usually suffices. If the sensor was operated in air, far more time must be factored in to drive out any residual oxygen from the dead volume, which is caused by the vessel design. Here, 2 hours applies as a general rule.

The zero point can be calibrated as soon as the sensor signal has settled. Here, the current measured value is calibrated to the zero value.

The reference method (sample calibration in zero point) can also be used here if suitable collecting vessels or a suitable reference measurement is available.

### 7.5.2 Zero point calibration with "zero solution"

Use oxygen-free water for this calibration. What is known as a "zero solution" is available from Endress+Hauser for oxygen calibration.

- As an alternative to the aqueous version, you can also work in an atmosphere that is free from oxygen, such as high-purity nitrogen.
- 1. Go to the "CAL/Oxygen/Zero point" menu.
- 2. Follow the instructions on the display.
- 3. Immerse the sensor in oxygen-free water or hold it in nitrogen (not in air!).
- 4. Accept the calibration data and then return to the measuring mode.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

### 7.5.3 Zero point calibration via data entry

- 1. Go to the "CAL/Oxygen/Zero point/Data input" menu.
- 2. Select "New zeropoint" and enter the new value.
- 3. Then select "OK" when you are asked to accept the calibration data for adjustment.
  - └ The new zero point is used.

# 7.6 Sample calibration

Calibration is possible both in the medium and in air. For this purpose, you measure the raw oxygen value using a reference measurement. You use this reference value to adjust the sensor. You can either calibrate the slope or the zero point with the reference value.

- 1. Go to the "CAL/Oxygen/Sample calibration" menu.
- 2. Choose between "Slope" and "Zero point".
  - Use the calibration of the zero point if you want to align the measurement with another measurement. You can correct the sensitivity of your measurement with the slope calibration.
- 3. Follow the instructions on the display.
- 4. Accept the calibration data and then return to the measuring mode.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

### 7.7 Resetting the counter

These functions do not adjust the sensor but reset the sensor's internal counters to "0".

**1** The counter for sensor cap calibrations is used to set warning limits and alarm limits for changing the membrane cap. This ensures that exhausted membrane caps are replaced in time.

### CAL/Oxygen

- 1. Select the desired function.
- 2. Follow the instructions.

Change electrolyte

- The internal sensor counter for calibrations with the electrolyte used is reset (not visible in the sensor information).
- Use this function after changing the electrolyte without replacing the membrane cap.

Change sensorcap

- The internal sensor counter for calibrations with the membrane cap used is reset. The number of calibrations performed with the current membrane cap is indicated in the sensor information.
- Select this function after replacing the membrane cap.

### 7.8 Temperature adjustment

- 1. Determine the temperature of your process medium with an alternative measurement, e.g. a precision thermometer.
- 2. Go to the "CAL/<Sensor type>/Temperature adjustment" menu.
- 3. Leave the sensor in the process medium and keep clicking "OK" until temperature measurement is started via the sensor.
- 4. Enter the reference temperature from the alternative measurement. You can either enter the absolute value or an offset for this purpose.
- 5. Once you have entered your data, click "OK" until the new data have been accepted.
  - └ This completes temperature sensor adjustment.
### 7.9 Error messages when performing the calibration

| Display message   | Causes and possible remedial measures   |
|---|---|
| The calibration is invalid. The range was overrun. Do you want repeat the last step?                    | Sensor contaminated or exhausted zero solution. As a result,<br>the permitted limit values for the zero point are exceeded<br>• Clean the sensor<br>• Renew the zero solution<br>• Repeat the calibration   |
| The stability criterion is not fulfilled. Do you want to repeat the last step?                          | <ul> <li>The measured value is not stable. As a result, the stability criterion is not met.</li> <li>Electrolyte and/or membrane cap exhausted, replace</li> <li>Adjust stability criteria (Menu/Setup/Inputs/Oxygen/Calib. settings/Stability criteria)</li> </ul> |
| Storage of the data failed. Do you want to retry?   | Optical sensor only!<br>The calibration data could not be stored in the sensor<br>• Check the sensor connection<br>• Repeat the calibration.  |
| Calibration aborted. Please clean sensor before<br>immersing in process medium. (Hold will be disabled) | The user has aborted the calibration.   |

## 8 Chlorine sensors

### 8.1 Calibration intervals

#### 8.1.1 Specifying the intervals

The calibration intervals depend heavily on:

- The application
- The sensor installation position

# You want to calibrate the sensor intermittently for a special application and/or on account of a special type of installation.

You can calculate the intervals using the following method:

- 1. Check the sensor:
  - a. Three months (potable water) or one month (process water) after commissioning the sensor
  - b. Using a reference measured value (DPD method) of a medium sample.
- 3. Compare the sensor measured value to the reference measured value.
- 4. Depending on your requirements, decide whether the deviation is acceptable or whether the sensor should be recalibrated.

Make sure to calibrate the sensor at least twice a year.

Please note that the DPD method itself is susceptible to high measured errors when the measured values are very low (< 0.2 mg/l) and can then no longer be regarded as a reliable method.

#### 8.1.2 Monitoring the calibration interval

Once you have established the calibration intervals for your process, you can also have the device monitor them.

#### Two functions are available to monitor the calibration interval:

- Calibration timer (Menu/Setup/Inputs/<Sensor type>/Extended setup/Calib. settings/Calibration timer)
  - You specify the calibration interval and the controller generates a diagnostics message once the set time has elapsed. You then recalibrate the sensor or replace it with a precalibrated sensor.

The timer is reset with the new calibration.

- Calibration validity (Menu/Setup/Inputs/<Sensor type>/Extended setup/Calib. settings/Calib. expiration date)
  - You set time limits to specify how long a calibration should be regarded as valid. Memosens sensors save all the calibration data. In this way it is easy to see whether the last calibration took place in the specified timeframe and is therefore still valid. This is particularly advantageous when working with precalibrated sensors.

#### 8.2 Polarization

The surface of the working electrode is polarized by the voltage applied by the transmitter between the cathode and anode. Therefore, after switching on the transmitter when a sensor is connected, you must wait until the polarization time elapses before starting the calibration. The sensor requires the following polarization times to obtain a stable display value:

Initial commissioning:

| CCS142D-A:      | 60 min. |
|-----------------|---------|
| CCS142D-G:      | 90 min. |
| Recommissioning |         |
| CCS142D-A:      | 30 min. |
| CCS142D-G:      | 45 min. |
|                 |         |

### 8.3 Types of calibration

The following types of calibration are possible:

- Slope
  - Sample calibration
  - Data entry
- Zero point
  - Sample calibration
  - Data entry
- Temperature adjustment

Furthermore, the calibration menu contains two additional functions to reset the sensor's internal counters:

- Change electrolyte
- Change sensorcap

### 8.4 Reference measurement

#### Reference measurement based on the DPD method

To calibrate the measuring system, perform a colorimetric comparison measurement based on the DPD method. Chlorine and chlorine dioxide react with diethyl-p-phenylenediamine (DPD) and turn red. The intensity of the red coloration is proportional to the chlorine content. This red coloration is measured with a photometer (e.g. CCM182) and indicated as the chlorine content.

#### Prerequisites

The sensor values are stable (no drift or fluctuating measured values for a period of at least 5 min). This is generally achieved by meeting the following requirements:

- You waited for the entire polarization time to elapse.
- The flow is allowable and constant.
- Temperature adjustment between the sensor and medium has occurred.
- The pH value is in the permitted range.

### 8.5 Slope calibration

#### 8.5.1 Sample calibration

You measure the raw chlorine value using a reference measurement. You use this reference value to adjust the sensor. You can either calibrate the slope or the zero point with the reference value.

- 1. Go to the "CAL/Chlorine menu.
- 2. Choose between: "Slope" and "Zero point".
  - Use the calibration of the zero point if you want to align the measurement with another measurement. You can correct the sensitivity of your measurement with the slope calibration.
- 3. Select "Sample calibration" and follow the instructions on the display.
- 4. Accept the calibration data and then return to the measuring mode.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

#### 8.5.2 Data entry

- 1. Go to the "CAL/Chlorine" menu.
- 2. Choose between: "Slope" and "Zero point".
  - Use the calibration of the zero point if you want to align the measurement with another measurement. You can correct the sensitivity of your measurement with the slope calibration.
- 3. Select "Data input" and enter the new value.
- 4. Accept the calibration data and then return to the measuring mode.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

### 8.6 Zero point calibration

Zero point calibration is particularly important if measurements are to be compared to one another, or in the event of measurements near the zero point.

A zero point shift in amperometric sensors is primarily caused by fouling on the cathode. The special mechanical construction of the sensor with the membrane cap and electrolyte almost completely eliminates such fouling.

#### 8.6.1 Sample calibration

You measure the raw chlorine value using a reference measurement. You use this reference value to adjust the sensor. You can either calibrate the slope or the zero point with the reference value.

- 1. Go to the "CAL/Chlorine menu.
- 2. Choose between: "Slope" and "Zero point".
  - Use the calibration of the zero point if you want to align the measurement with another measurement. You can correct the sensitivity of your measurement with the slope calibration.
- 3. Select "Sample calibration" and follow the instructions on the display.
- 4. Accept the calibration data and then return to the measuring mode.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

#### 8.6.2 Zero point calibration via data entry

- 1. Go to the "CAL/Chlorine" menu.
- 2. Choose between: "Slope" and "Zero point".
  - Use the calibration of the zero point if you want to align the measurement with another measurement. You can correct the sensitivity of your measurement with the slope calibration.
- 3. Select "Data input" and enter the new value.
- 4. Accept the calibration data and then return to the measuring mode.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

#### 8.7 Resetting the counter

These functions do not adjust the sensor but reset the sensor's internal counters to "O".

The counter for sensor cap calibrations is used to set warning limits and alarm limits for changing the membrane cap. This ensures that exhausted membrane caps are replaced in time.

#### CAL/Chlorine

- 1. Select the desired function.
- 2. Follow the instructions.

Change electrolyte

- The internal sensor counter for calibrations with the electrolyte used is reset (not visible in the sensor information).
- Use this function after changing the electrolyte without replacing the membrane cap.

Change sensorcap

- The internal sensor counter for calibrations with the membrane cap used is reset. The number of calibrations performed with the current membrane cap is indicated in the sensor information.
- Select this function after replacing the membrane cap.

#### 8.8 Temperature adjustment

- 1. Determine the temperature of your process medium with an alternative measurement, e.g. a precision thermometer.
- 2. Go to the "CAL/<Sensor type>/Temperature adjustment" menu.
- 3. Leave the sensor in the process medium and keep clicking "OK" until temperature measurement is started via the sensor.
- 4. Enter the reference temperature from the alternative measurement. You can either enter the absolute value or an offset for this purpose.
- 5. Once you have entered your data, click "OK" until the new data have been accepted.
  - └ This completes temperature sensor adjustment.

### 8.9 Error messages when performing the calibration

| Display message   | Causes and possible remedial measures   |
|---|---|
| The calibration is invalid. The range was overrun. Do you want repeat the last step?                    | Sensor contaminated. As a result, the permitted limit values<br>for the zero point are exceeded<br>• Clean the sensor<br>• Repeat the calibration   |
| The stability criterion is not fulfilled. Do you want to repeat the last step?                          | <ul> <li>The measured value is not stable. As a result, the stability criterion is not met.</li> <li>Electrolyte and/or membrane cap exhausted, replace</li> <li>Adjust stability criteria<br/>(Menu/Setup/Inputs/Chlorine/Calib. settings/Stability criteria)</li> </ul> |
| Calibration aborted. Please clean sensor before<br>immersing in process medium. (Hold will be disabled) | The user has aborted the calibration.   |

## 9 Ion-selective sensors

Some measured values from other electrodes or sensors are used for measured value compensation of ion-selective electrodes:

- Measured value of temperature sensor for temperature compensation
- pH measured value for pH compensation of ammonium (optional)
- Potassium or chloride measured value for compensation of interference ions in the case of ammonium or nitrate (optional)

For this reason, there is a sequence for calibration and adjustment that must be followed in order to achieve a reliable measurement:

- 1. Temperature adjustment
- 2. Calibration and adjustment of pH electrode
- 3. Depending on whether compensation electrodes are used:
  - Calibration and adjustment of ion-selective compensation electrodes (potassium, chloride)
  - If no compensation electrodes are used:
    - A correct manual offset is configured for the ammonium and nitrate electrode
- 4. Calibration and adjustment of ion-selective measuring electrodes (ammonium, nitrate)

### 9.1 Types of calibration

The following types of calibration are possible:

- pH electrode:
  - Two-point calibration
  - Single-point calibration
- Ion-selective electrodes:
  - Single-point calibration
  - Data entry
  - Two-point calibration
  - Standard addition ("Expert" only)
  - Sample calibration ("Expert" only)
- ORP sensor:
  - Single-point calibration
- Temperature adjustment by entering a reference value

## 9.2 pH

#### 9.2.1 Two-point calibration

You use calibration buffers to perform two-point calibration. The quality buffers supplied by Endress+Hauser are certified and measured in an accredited laboratory. The accreditation (DAR registration number "DKD-K-52701") confirms that the actual values and the maximum deviations are correct and traceable.

- 1. Go to the "CAL/ISE/CAL/2-pnt. calibration" menu.
- 2. Follow the instructions on the display.
- 3. Press "OK" after you have immersed the sensor into the first buffer.
  - ← The system starts calculating the measured value for the first buffer. Once the stability criterion is met, the measured value is displayed in mV.
- 4. Continue to follow the instructions.
- 5. Press "OK" **after** you have immersed the sensor into the second buffer.
  - The system starts calculating the measured value for the buffer. Once the stability criterion is met, the measured values of the two buffers and the calculated values for the slope and zero point are displayed.
- 6. Select "OK" when you are asked to accept the calibration data for adjustment.
- 7. Put the sensor back into the medium and press "OK" again.
  - └ This deactivates the hold and the system starts measuring again.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

**Only use calibration buffers once.** 

#### 9.2.2 Single-point calibration

- 1. Go to the "CAL/ISE/1-pnt. calibration" menu.
- 2. Select the pH electrode and start the calibration.
- 3. Specify the measured value from the reference measurement.
- 4. Follow the instructions and immerse the sensor into the buffer.
- 5. Start the calibration.
- 6. Select "OK" when you are asked to accept the calibration data for adjustment.
- 7. Put the sensor back into the medium and press "OK" again.
  - └ This deactivates the hold and the system starts measuring again.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

Only use calibration buffers once.

### 9.3 Ammonium, nitrate, potassium, chloride

In the case of potentiometric methods to determine the ion concentration, the voltage from the electrochemical measuring cell, comprising the ion-selective electrode and a reference electrode, is proportional to the logarithm of the concentration (or activity) of the ions under analysis within the "linear" or "NERNST" range . The calibration parameters slope and zero point are based on this logarithmic relation, giving these parameters a completely different meaning here than in other measurement methods.

#### 9.3.1 Single-point calibration

You can calibrate up to 2 ion-selective electrodes simultaneously (not pH/ORP).

Simply select the electrodes to be calibrated from the list.

You use a calibration solution with a known concentration.

- 1. Go to the "CAL" menu and select the electrode to be calibrated.
- 2. Select the "1-pnt. calibration" type of calibration.
- 3. Immerse the sensor into the calibration solution and follow the instructions on the display.
- 4. Enter the concentration of the calibration solution and follow the instructions.
- 5. Accept the calibration data and then return to the measuring mode.
  - └ This deactivates the hold and the system starts measuring again.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

During the calibration, move the sensor in the tank to ensure the necessary flow of medium to the ion-selective electrode.

#### 9.3.2 Two-point calibration

Remove the sensor from the medium for calibration.

- 1. Go to the "CAL" menu and select the electrode to be calibrated.
- 2. Select the "2-pnt. calibration" type of calibration.
- 3. Follow the instructions on the display.
- 4. Press "OK" after you have immersed the sensor into the first calibration solution.
  - The sensor starts calculating the measured value. Once the stability criterion is met, the measured value is displayed.
- 5. Continue to follow the instructions.
- 6. Press "OK" after you have immersed the sensor into the second calibration solution.
  - The sensor starts calculating the measured value. Once the stability criterion is met, the measured values of the two calibration solutions and the calculated values for the slope and zero point are displayed.
- 7. Select "OK" when you are asked to accept the calibration data for adjustment.
- 8. Put the sensor back into the medium and press "OK" again.
  - ← This deactivates the hold and the system starts measuring again.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

During the calibration, move the sensor in the tank to ensure the necessary flow of medium to the ion-selective electrode.

#### 9.3.3 Data entry

You enter the slope and zero point manually. The calibration function is calculated from these values. Thus, the data entry returns the same result as two-point calibration. You must determine the slope and zero point in an alternative way.

- 1. Go to the "CAL" menu and select the electrode to be calibrated.
- 2. Select the "Data input" type of calibration.
  - ← The slope and zero point are shown on the display.
- 3. Select each value one after another and then enter your desired numerical value.
  - Since you are entering all the variables directly, no additional information is displayed by the controller.

You can cancel the calibration any time by pressing the "ESC" key. No new data are then used to adjust the sensor.

### 9.4 ORP

- With this type of calibration, you work with calibration buffers, e.g. ORP buffers from Endress+Hauser. For this calibration, you remove the sensor from the medium.
- 1. Go to the "CAL/ISE/Redox/1-pnt. calibration" menu.
- 2. Follow the instructions on the display.
- 3. Accept the calibration data and then return to the measuring mode.
  - └ This deactivates the hold and the system starts measuring again.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

### 9.5 Temperature adjustment

- 1. Determine the temperature of your process medium with an alternative measurement, e.g. a precision thermometer.
- 2. Go to the "CAL/<Sensor type>/Temperature adjustment" menu.
- 3. Leave the sensor in the process medium and keep clicking "OK" until temperature measurement is started via the sensor.
- 4. Enter the reference temperature from the alternative measurement. You can either enter the absolute value or an offset for this purpose.
- 5. Once you have entered your data, click "OK" until the new data have been accepted.
  - └ This completes temperature sensor adjustment.

### 9.6 Error messages when performing the calibration

| Display message  | Causes and possible remedial measures  |
|--|--|
| The calibration is invalid. Do you want to start a new calibration?<br>Slope out of tolerance.<br>Zeropoint out of tolerance.<br>Sample concentration too low. | <ul> <li>The calibration buffer is contaminated or the pH value is no longer within the permitted limits. As a result, the permitted measured value deviation is exceeded.</li> <li>Check the expiry date</li> <li>Use a fresh buffer</li> </ul>   |
|  | <ul> <li>Incorrect buffers used. As a result, the buffer recognition function - for example - does not work correctly.</li> <li>pH values of the buffers are too close together, e.g. pH 9 and 9.2</li> <li>Use buffers with a larger pH difference</li> </ul>   |
|  | Sensor aging or contaminated. As a result, the permitted limit<br>values for the slope and/or zero point are exceeded<br>• Clean the sensor<br>• Adjust the limit values<br>• Regenerate or replace the sensor   |
| The stability criterion is not fulfilled. Do you want to repeat the last step?   | The measured value or temperature is not stable. As a result,<br>the stability criterion is not met.<br>• Keep the temperature constant during calibration<br>• Replace the buffer<br>• Sensor old or contaminated. Clean or regenerate.<br>• Adjust the stability criteria<br>(Menu/Setup/Inputs/ <electrode slot="">/Calib.<br/>settings/Stability criteria)</electrode> |
| Calibration aborted. Please clean sensor before<br>immersing in process medium. (Hold will be disabled)  | The user has aborted the calibration.  |

## 10 Turbidity and solids sensor

The sensor facilitates measurements with different methods adapted to suit the measuring tasks. The method is specified by selecting the appropriate application and the reference model.

More information on the models and methods available is provided in the Operating Instructions of the sensor.

### 10.1 Types of calibration

In addition to the uneditable factory calibration, the sensor stores five other data records. Each calibration data record can have up to five calibration points.

- Single-point calibration This causes a change in the slope. This type of calibration is used if the measured value only changes to a limited extent.
- Two-point calibration This causes a change in the slope and zero point. This type of calibration is used if the measured value changes to a large extent.
- Multipoint calibration
   Calibration at three or more points always causes the measuring curve to be recalculated (zero point and slope).
- Temperature adjustment via reference value

Single-point and two-point calibration are based on the data record stored internally in the device.

The **three-point calibration** of the sensor is the standard calibration.

It is absolutely essential:

- When commissioning the sensor in sludge applications
- When measuring in another type of sludge

Three-point calibration of the sensor is **not necessary** when recalibrating with the same type of sludge. Single point calibration suffices here if the levels of turbidity do not differ too much.

### 10.2 Turbidity and solids

#### 10.2.1 Factory calibration

The sensor is precalibrated on leaving the factory. As such, it can be used in a wide range of applications (e.g. clear water measurement) without the need for additional calibration. The factory calibration is based on a three-point calibration of a reference sample.

The factory calibration cannot be deleted and can be retrieved at any time. All other calibrations  $\ -$ 

performed as customized calibrations - are referenced to this factory calibration.

#### 10.2.2 Calibration and adjustment principle

The calibration is always based on a factory calibration saved.

If you use one or two medium concentration values for calibration, the factory data record is recalculated using these measuring points (non-linear function) and saved as the **new data record**. The original factory calibration is not lost.

If you use three or more concentration values for calibration, a completely new calibration function is calculated that no longer takes the original data record into account.

Give your calibration data records meaningful and useful names. For example, the name could contain the name of the application on which your data record was originally based. This makes it easier for you to distinguish between different data records.

#### 10.2.3 Determining the reference value in the laboratory

- 1. Take a representative sample of the medium.
- 2. Make sure the sample is as homogeneous as possible.
- 3. Determine the solids content or the turbidity of the sample with the laboratory method.
- 4. Use the laboratory measured value as the reference value for calibrating the sensor.
- You can also calibrate with samples with added concentrations, or where solid particles have settled to the base of the tank. In this way, you use a serial dilution to obtain calibration points that are above and below the expected turbidity or the expected solids content.

#### 10.2.4 Calibration and adjustment of the sensor

Use the same medium sample that you used to determine the laboratory measured value.

- 1. Make sure the sample is as homogeneous as possible.
- 2. Create the necessary number of calibration samples by diluting the medium sample into suitable concentrations. For example, you generally obtain very good calibration results with a three-point calibration with concentrations levels of 100 : 33 : 10.



Fig. 8: Creating samples for a three-point calibration

A Original sample

B 1 part sample A + 2 parts water

C 1 part sample A + 9 parts water

- 3. Determine the solids content or the turbidity of the calibration samples in a series of decreasing concentrations.
- 4. Calculate the reference values of your serial dilution from the laboratory measured value and enter these for every calibration point.

#### 1. Sequence of menus during the calibration

- a. Menu/Setup/Inputs/Turbidity/Application Select the application whose saved calibration function is to be changed by additional measuring points.
- b. CAL/Turbidity/Assay Select a data record for your serial dilution.
- c. Dataset name Assign a name to the data record.
- Basic application
   Select the same application as selected in a.
- Unit Decide which unit you want to use. Use the unit in which you also obtained the laboratory values.
- 2. First measuring point (lowest concentration)
  - a. Follow the instructions on the display.
  - b. Once a stable measured value has been determined, you will be asked for the set point (=laboratory value) of the sample. Enter this set point.
- 3. Decide:
  - a. Whether you want to add another value (next highest concentration) to your data record ("Calibrate next assay") or
  - b. Whether you want to end the calibration and accept the data for adjustment ("Take over the calibration data?").
- 4. Determine all the desired measuring points as explained in Steps 2 and 3.
- 5. Completing the calibration and adjustment
  - a. Once you have determined the last measuring point, accept the data. A message is displayed informing you whether the data record is valid.
  - b. Follow the instructions and then press "OK".
  - c. You are also asked whether you want to activate the data record just recorded. If you select "OK", the measured values are calculated on the basis of the new calibration function.
  - d. You still have the possibility of editing the data record. Once you have activated the data record you can only change the set points. It is then no longer possible to delete measuring points.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

#### 10.2.5 Duplicating data records

This function makes it possible to edit an existing calibration data record, such as the factory calibration. By entering appropriate data you can then configure an offset for the copied data record or change the nominal values using a table. This provides a quick and easy way to react to modified conditions in your process which you are aware of without the need for calibration.

- 1. Start the function "Duplicate dataset".
- 2. Select the data record which you want to duplicate.
- 3. Then select the storage location and give a name to the duplicated record.
  - └→ You can only duplicate a record if you have not yet used all the available space for data records. If no more room is available you must first delete a data record.
- 4. You can then configure an offset for the new data record or change the nominal values of the individual calibration points via the "Edit table" function.
- 5. If you want to use the modified data record, you then have to go to the Setup/Inputs menu and select the new data record under "Application".

### 10.3 Temperature adjustment

- 1. Determine the temperature of your process medium with an alternative measurement, e.g. a precision thermometer.
- 2. Go to the "CAL/<Sensor type>/Temperature adjustment" menu.
- 3. Leave the sensor in the process medium and keep clicking "OK" until temperature measurement is started via the sensor.
- 4. Enter the reference temperature from the alternative measurement. You can either enter the absolute value or an offset for this purpose.
- 5. Once you have entered your data, click "OK" until the new data have been accepted.
  - └ This completes temperature sensor adjustment.

### 10.4 Error messages when performing the calibration

| Display message  | Causes and possible remedial measures   |
|--|---|
| The stability criterion is not fulfilled. Do you want to repeat the last step?                       | <ul> <li>The measured value or temperature is not stable. As a result, the stability criterion is not met.</li> <li>Keep the temperature constant during calibration</li> <li>Sensor old or contaminated. Clean or regenerate.</li> <li>Adjust the stability criteria (Menu/Setup/Inputs<sensor type="">Calib. settings/Stability criteria)</sensor></li> </ul> |
| The calibrated dataset is invalid. Do you want to restart the calibration?                           | Calibration point not plausible<br>Repeat the calibration<br>Exchange the calibration medium<br>Sensor fouled> Clean  |
| Calibration aborted. Please clean sensor before immersing in process medium. (Hold will be disabled) | The user has aborted the calibration.   |

## 11 SAC sensor

### 11.1 Types of calibration

In addition to the uneditable factory calibration, the sensor stores six other data records. Each calibration data record can have up to five calibration points.

- Single-point calibration This causes a change in the slope. This type of calibration is used if the measured value only changes to a limited extent.
- Two-point calibration
   This causes a change in the slope and zero point. This type of calibration is used if the measured value changes to a large extent.
- Multipoint calibration Calibration at three or more points always causes the measuring curve to be recalculated (zero point and slope).
- Temperature adjustment via reference value

Single-point and two-point calibration are based on the data record stored internally in the device.

### 11.2 SAC

#### 11.2.1 Factory calibration

The sensor is precalibrated on leaving the factory. As a nitrate sensor, it can be used in a wide range of clear water measurements without the need for additional calibration. In the case of a SAC sensor, it is advantageous to calibrate to the customer's specific process in the majority of cases. The factory calibration is based on a three-point calibration of a reference sample. The factory calibration cannot be deleted and can be retrieved at any time. All other calibrations

performed as customized calibrations - are referenced to this factory calibration.

#### 11.2.2 Principle of calibration

The calibration is always based on a factory calibration saved.

If you use one or two medium concentration values for calibration, the factory data record is recalculated using these measuring points (non-linear function) and saved as the **new data record**. The original factory calibration is not lost.

If you use three or more concentration values for calibration, a completely new calibration function is calculated that no longer takes the original data record into account.

Give your calibration data records meaningful and useful names. For example, the name could contain the name of the application on which your data record was originally based. This makes it easier for you to distinguish between different data records.

#### **11.2.3** Determining the reference values in the laboratory

Different methods are available for the calibration:

- Serial dilution of a medium sample
- Serial dilution with standard solutions (KHP=potassium hydrogen phthalate)
- Combination of both (medium sample with added standard)
- 1. Take a representative sample of the medium.
  - └ Outlet water, for example, is very suitable for obtaining a representative sample. In this case, you do not have to carry out the subsequent step concerning value stabilization.
- 2. Take suitable measures to ensure that the process of biological and chemical reduction in the sample does not progress any further.
- 3. Determine the measured values of your sample array with the laboratory method (e.g. by colorimetric means using a cuvette test).

#### 11.2.4 Calibration and adjustment of the sensor

To calibrate the sensor, use the same medium sample or sample array that you used to determine the laboratory measured values. The sample array can also be pure standard solutions.

Proceed as follows depending on how many measuring points you want to determine for the calibration:

- 1. Calibrate the sensor with the first measuring point and enter the laboratory measured value as the reference value.
- 2. If you only want to calibrate one point end the calibration by accepting the calibration data.
  - └ → Otherwise continue with the next step.
- 3. Add parent solution to the sample for the 2nd measuring point and determine the measured value. The reference value is calculated from the laboratory measured value plus the added concentration.
- 4. Repeat Step b as often as needed until you have the desired number of calibration points (max. 5).

To avoid incorrect calibration from carryover:

- Always go from a low concentration to a high concentration.
- Clean and dry the sensor after each measurement.
- Make sure to remove medium residue in the sensor gap and in the connection opening for the compressed air (e.g. by rinsing with the next calibration solution).

#### 1. Menu settings for the calibration

a. CAL/SAC/Assay

Select a data record for your serial dilution.

- b. Dataset name Assign a name to the data record.
- c. Basic application Decide which value you want to calibrate: SAC, COD, TOC, DOC or BOD.
- d. Unit

Decide which unit you want to use. Use the unit in which you also obtained the laboratory values.

- e. Only for: Basic application = "SAC" The transmitter can determine the derived variables COD, TOC, DOC and BOD from the SAC value. Different calculation factors exist for this depending on the reference method. You can adapt the calculation factor saved at the factory for COD/BOD and TOC/DOC to your application and also enter an SAC offset.
- 2. First measuring point (lowest concentration)
  - a. Start the calibration and follow the instructions on the display.
  - b. Once a stable measured value has been determined, you will be asked for the set point (=laboratory value) of the sample. Enter this set point.
- 3. Decide:
  - a. Whether you want to add another value (next highest concentration) to your data record ("Calibrate next assay") or
  - b. Whether you want to end the calibration and accept the data for adjustment ("Take over the calibration data?").
- 4. Determine all the desired measuring points as explained in Steps 2 and 3.
- 5. Completing the calibration and adjustment
  - a. Once you have determined the last measuring point, accept the data. A message is displayed informing you whether the data record is valid.
  - b. Select "OK" when you are asked to accept the calibration data for adjustment.
  - c. You are then asked whether you want to activate the data record just recorded. If you select "OK", the measured values are calculated on the basis of the new calibration function.
  - d. You still have the possibility of editing the data record. Once you have activated the data record you can only change the set points. It is then no longer possible to delete measuring points.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

#### 11.2.5 Duplicating data records

This function makes it possible to edit an existing calibration data record, such as the factory calibration. By entering appropriate data you can then configure an offset for the copied data record or change the nominal values using a table. This provides a quick and easy way to react to modified conditions in your process which you are aware of without the need for calibration.

- 1. Start the function "Duplicate dataset".
- 2. Select the data record which you want to duplicate.
- 3. Then select the storage location and give a name to the duplicated record.
  - └→ You can only duplicate a record if you have not yet used all the available space for data records. If no more room is available you must first delete a data record.
- 4. You can then configure an offset for the new data record or change the nominal values of the individual calibration points via the "Edit table" function.
- 5. If you want to use the modified data record, you then have to go to the Setup/Inputs menu and select the new data record under "Application".

### 11.3 Temperature adjustment

- 1. Determine the temperature of your process medium with an alternative measurement, e.g. a precision thermometer.
- 2. Go to the "CAL/<Sensor type>/Temperature adjustment" menu.
- 3. Leave the sensor in the process medium and keep clicking "OK" until temperature measurement is started via the sensor.
- 4. Enter the reference temperature from the alternative measurement. You can either enter the absolute value or an offset for this purpose.
- 5. Once you have entered your data, click "OK" until the new data have been accepted.
  - └ This completes temperature sensor adjustment.

### 11.4 Error messages when performing the calibration

| Display message   | Causes and possible remedial measures   |
|---|---|
| The stability criterion is not fulfilled. Do you want to repeat the last step?                          | <ul> <li>The measured value or temperature is not stable. As a result, the stability criterion is not met.</li> <li>Keep the temperature constant during calibration</li> <li>Sensor old or contaminated. Clean or regenerate.</li> <li>Adjust the stability criteria (Menu/Setup/Inputs<sensor type="">Calib. settings/Stability criteria)</sensor></li> </ul> |
| The calibrated dataset is invalid. Do you want to restart the calibration?                              | Calibration point not plausible<br>Repeat the calibration<br>Exchange the calibration medium<br>Sensor fouled> Clean  |
| Calibration aborted. Please clean sensor before<br>immersing in process medium. (Hold will be disabled) | The user has aborted the calibration.   |

## 12 Nitrate sensors

Calibration is performed in the process by comparing the values to an external standard method, by calibrating with standard solutions or by using a combination of both.

- Processes with high nitrate values (> 0.1 mg/l) Take a sample and determine the nitrate concentration in the laboratory. Then calibrate and adjust the sensor with the laboratory value.
- Processes with very different nitrate values At time A, take a sample with a high concentration, and measure and calibrate the sample. At time B - which can be a few days after time A - take a sample with a low concentration, and measure and calibrate the second value.
- Calibration with the addition of standard
   If the sludge parameters tend to be constant, you can perform the calibration with a sample with a low concentration of nitrate and then add standard to the sample.
   Take a larger sample (bucket) and analyze some of it by colorimetric means. Calibrate this value in the sensor. Then add standard to the sample, determine the laboratory value and calibrate the value in the sensor.

#### Additional calibration points, recalibration

Points can be added to an existing calibration (max. 5 points per data record). In this way, different media or concentrations can be included in the calibration at different times.

### 12.1 Types of calibration

In addition to the uneditable factory calibration, the sensor stores six other data records. Each calibration data record can have up to five calibration points.

- Single-point calibration This causes a change in the slope. This type of calibration is used if the measured value only changes to a limited extent.
- Two-point calibration This causes a change in the slope and zero point. This type of calibration is used if the measured value changes to a large extent.
- Multipoint calibration Calibration at three or more points always causes the measuring curve to be recalculated (zero point and slope).
- Temperature adjustment via reference value

Single-point and two-point calibration are based on the data record stored internally in the device.

### 12.2 Nitrate

#### 12.2.1 Factory calibration

The sensor is precalibrated on leaving the factory. As a nitrate sensor, it can be used in a wide range of clear water measurements without the need for additional calibration. In the case of a SAC sensor, it is advantageous to calibrate to the customer's specific process in the majority of cases. The factory calibration is based on a three-point calibration of a reference sample. The factory calibration cannot be deleted and can be retrieved at any time. All other calibrations - performed as customized calibrations - are referenced to this factory calibration.

#### 12.2.2 Principle of calibration

The calibration is always based on a factory calibration saved.

If you use one or two medium concentration values for calibration, the factory data record is recalculated using these measuring points (non-linear function) and saved as the **new data record**. The original factory calibration is not lost.

If you use three or more concentration values for calibration, a completely new calibration function is calculated that no longer takes the original data record into account.

Give your calibration data records meaningful and useful names. For example, the name could contain the name of the application on which your data record was originally based. This makes it easier for you to distinguish between different data records.

#### 12.2.3 Determining the reference values in the laboratory

- 1. Take a representative sample of the medium.
  - Outlet water is very suitable for obtaining a representative sample. In this case, you do
    not have to carry out the subsequent step concerning value stabilization.
- 2. Take suitable measures to ensure that the process of nitrate reduction in the sample does not progress any further, such as immediate filtration (0.45  $\mu$ m) of the sample as per DIN 38402.
- 3. Determine the concentration of nitrate in the sample with the laboratory method (e.g. determining the concentration by colorimetric means using a cuvette test standard method in accordance with DIN 38405 Part 9).

#### 12.2.4 Calibration and adjustment of the sensor

To calibrate the sensor, use the same medium sample or sample array that you used to determine the laboratory measured values. The sample array can also be pure standard solutions.

Proceed as follows depending on how many measuring points you want to determine for the calibration:

- 1. Calibrate the sensor with the first measuring point and enter the laboratory measured value as the reference value.
  - └ If you only want to calibrate one point end the calibration by accepting the calibration data. Otherwise continue with the next step.
- 2. Add parent solution to the sample for the 2nd measuring point and determine the measured value. The reference value is calculated from the laboratory measured value plus the added concentration.
- 3. Repeat Step b as often as needed until you have the desired number of calibration points (max. 5).

To avoid incorrect calibration from nitrate carryover:

- Always go from a low concentration to a high concentration.
- Clean and dry the sensor after each measurement.
- Make sure to remove medium residue in the sensor gap and in the connection opening for the compressed air (e.g. by rinsing with the next calibration solution).

#### 1. Menu settings for the calibration

- a. CAL/Nitrate/Assay
  - Select a data record for your serial dilution.
- b. Dataset name Assign a name to the data record.
- c. Unit

Decide which unit you want to use. Use the unit in which you also obtained the laboratory values.

- 2. First measuring point (lowest concentration)
  - a. Follow the instructions on the display.
  - b. Once a stable measured value has been determined, you will be asked for the set point (=laboratory value) of the sample. Enter this set point.
- 3. Decide:
  - a. Whether you want to add another value (next highest concentration) to your data record ("Calibrate next assay") or
  - b. Whether you want to end the calibration and accept the data for adjustment ("Take over the calibration data?").
- 4. Determine all the desired measuring points as explained in Steps 2 and 3.

- 5. Completing the calibration and adjustment
  - a. Once you have determined the last measuring point, accept the data. A message is displayed informing you whether the data record is valid.
  - b. Select "OK" when you are asked to accept the calibration data for adjustment.
  - c. You are then asked whether you want to activate the data record just recorded. If you select "OK", the measured values are calculated on the basis of the new calibration function.
  - d. You still have the possibility of editing the data record. Once you have activated the data record you can only change the set points. It is then no longer possible to delete measuring points.

You can cancel the calibration any time by pressing the "ESC" key. No data are then used to adjust the sensor.

#### 12.2.5 Duplicating data records

This function makes it possible to edit an existing calibration data record, such as the factory calibration. By entering appropriate data you can then configure an offset for the copied data record or change the nominal values using a table. This provides a quick and easy way to react to modified conditions in your process which you are aware of without the need for calibration.

- 1. Start the function "Duplicate dataset".
- 2. Select the data record which you want to duplicate.
- 3. Then select the storage location and give a name to the duplicated record.
  - └→ You can only duplicate a record if you have not yet used all the available space for data records. If no more room is available you must first delete a data record.
- 4. You can then configure an offset for the new data record or change the nominal values of the individual calibration points via the "Edit table" function.
- 5. If you want to use the modified data record, you then have to go to the Setup/Inputs menu and select the new data record under "Application".

#### 12.3 Temperature adjustment

- 1. Determine the temperature of your process medium with an alternative measurement, e.g. a precision thermometer.
- 2. Go to the "CAL/<Sensor type>/Temperature adjustment" menu.
- 3. Leave the sensor in the process medium and keep clicking "OK" until temperature measurement is started via the sensor.
- 4. Enter the reference temperature from the alternative measurement. You can either enter the absolute value or an offset for this purpose.
- 5. Once you have entered your data, click "OK" until the new data have been accepted.
  - └ This completes temperature sensor adjustment.

### 12.4 Error messages when performing the calibration

| Display message  | Causes and possible remedial measures   |
|--|---|
| The stability criterion is not fulfilled. Do you want to repeat the last step?                       | <ul> <li>The measured value or temperature is not stable. As a result, the stability criterion is not met.</li> <li>Keep the temperature constant during calibration</li> <li>Sensor old or contaminated. Clean or regenerate.</li> <li>Adjust the stability criteria (Menu/Setup/Inputs<sensor type="">Calib. settings/Stability criteria)</sensor></li> </ul> |
| The calibrated dataset is invalid. Do you want to restart the calibration?                           | Calibration point not plausible<br>Repeat the calibration<br>Exchange the calibration medium<br>Sensor fouled> Clean  |
| Calibration aborted. Please clean sensor before immersing in process medium. (Hold will be disabled) | The user has aborted the calibration.   |

## 13 Calibration accessories

### 13.1 pH calibration buffer

#### Quality buffers from Endress+Hauser

Solutions which are traced by the DAkkS-accredited Endress+Hauser buffer laboratory (DAkkS = German accreditation body) to primary reference material of the PTB and to standard reference material of the National Institute of Standards and Technology (NIST) in accordance with DIN 19266 are used as secondary reference buffer solutions.

|        | pH | value | lue   |        |                             |  |  |  |  |
|--------|----|-------|---|--------|-----------------------------|--|--|--|--|
|        | А  | pH 2  | $H 2.00 (accuracy \pm 0.02 pH)$                         |        |                             |  |  |  |  |
|        | С  | pH 4  | $hH 4.00 (accuracy \pm 0.02 \text{ pH})$                |        |                             |  |  |  |  |
|        | Е  | pH 🛛  | 7.00  | (accu  | racy ± 0.02 pH)             |  |  |  |  |
|        | G  | pH 9  | 9.00  | (accu  | $aracy \pm 0.02 \text{ pH}$ |  |  |  |  |
|        | Ι  | pH 9  | 9.20  | (accu  | $racy \pm 0.02 \text{ pH}$  |  |  |  |  |
|        | К  | pH 1  | 10.00   | ) (ac  | curacy $\pm 0.05$ pH)       |  |  |  |  |
|        | Μ  | pH :  | 12.00   | ) (aco | curacy $\pm 0.05$ pH)       |  |  |  |  |
|        |    | Qua   | Quantity  |        |                             |  |  |  |  |
|        |    | 01    | 01 20 x 18 ml (0.68 fl.oz) only buffer pH 4.00 and 7.00 |        |                             |  |  |  |  |
|        |    | 02    | 02 250 ml (8.45 fl.oz)                                  |        |                             |  |  |  |  |
|        |    | 10    | 10 1000 ml (0.26 US gal)                                |        |                             |  |  |  |  |
|        |    | 50    | 50 5000 ml (1.32 US gal) canister for Topcal S          |        |                             |  |  |  |  |
|        |    |       | Certificate   |        |                             |  |  |  |  |
|        |    |       | A Buffer analysis certificate                           |        |                             |  |  |  |  |
|        |    |       | Version   |        |                             |  |  |  |  |
|        |    |       |   | 1      | Standard                    |  |  |  |  |
| CPY20- |    |       |   |        | Complete order code         |  |  |  |  |

### 13.2 ORP buffer

Technical ORP buffer solutions

- +220 mV, pH 7, 100 ml (3.4 fl.oz.); Order No. CPY3-0
- +468 mV, pH 0.1, 100 ml (3.4 fl.oz.); Order No. CPY3-1

### 13.3 Conductivity

#### **Calibration solutions**

Precision solutions referenced to the NIST standard reference material (SRM) for the qualified calibration of conductivity measuring systems as per ISO, accuracy  $\pm$  0.5 %, with temperature table,

- CLY 11-A
   74.0 μS/cm (reference temperature 25 °C), 500 ml
   Order No. 50081902
- CLY 11-B 149.6 μS/cm (reference temperature 25 °C), 500 ml Order No. 50081903
- CLY 11-C 1.406 mS/cm (reference temperature 25 °C), 500 ml Order No. 50081904
- CLY 11-D 12.64 mS/cm (reference temperature 25 °C), 500 ml Order No. 50081905
- CLY 11-E 107.00 mS/cm (reference temperature 25 °C), 500 ml Order No. 50081906

### 13.4 Oxygen

#### 13.4.1 Zero solution

- 3 screw-cap bottles for producing 3 x 1 liter oxygen-free solution
- Order No. 50001041

#### 13.4.2 Calibration vessel

Calibration vessel

- for COS61/61D
- Order No.: 51518599

### 13.5 Chlorine

CCM182

- Microprocessor-controlled photometer for the determination of chlorine and pH value
- Chlorine measuring range: 0.05 6 mg/l
- pH value measuring range: 6.5 8.4
- Order No.: CCM182-0

### 13.6 ISE and nitrate

|        | Star | tandard solution |                           |                    |                            |  |  |  |  |  |
|--------|------|------------------|---------------------------|--------------------|----------------------------|--|--|--|--|--|
|        | 1    | Am               | mmonium nitrate, 1 mole   |                    |                            |  |  |  |  |  |
|        | 2    | Pota             | otassium chloride, 1 mole |                    |                            |  |  |  |  |  |
|        |      | Con              | taine                     | er siz             | ze                         |  |  |  |  |  |
|        |      | А                | 250 ml (8.45 fl.oz.)      |                    |                            |  |  |  |  |  |
|        |      |                  | Tra                       | ransport documents |                            |  |  |  |  |  |
|        |      |                  | 1                         | Star               | ndard documents            |  |  |  |  |  |
|        |      |                  | 2                         | Incl               | dangerous goods sheets     |  |  |  |  |  |
|        |      |                  | 3                         | Safety data sheet  |                            |  |  |  |  |  |
|        |      |                  |                           | Certificate        |                            |  |  |  |  |  |
|        |      |                  |                           | А                  | None                       |  |  |  |  |  |
|        |      |                  |                           | В                  | Manufacturer's certificate |  |  |  |  |  |
| CAY40- |      |                  |                           |                    | Complete order code        |  |  |  |  |  |

### 13.7 Nitrate

Nitrate standard solutions, 1 liter

- Standard 5 mg/l NO<sub>3</sub> N; Order No. CAY342-V10C05AAE
- Standard 10 mg/l NO<sub>3</sub> N; Order No. CAY342-V10C10AAE
- Standard 15 mg/l NO<sub>3</sub> N; Order No. CAY342-V10C15AAE
- Standard 20 mg/l NO<sub>3</sub> N; Order No. CAY342-V10C20AAE
- Standard 30 mg/l NO<sub>3</sub> N; Order No. CAY342-V10C30AAE
- Standard 40 mg/l NO<sub>3</sub> N; Order No. CAY342-V10C40AAE
- Standard 50 mg/l NO<sub>3</sub> N; Order No. CAY342-V10C50AAE

### 13.8 SAC

KHP standard solution

• CAY451-V10C01AAE, 1000 ml parent solution 5 000 mg/l TOC

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