Combined dCa ISE

6.00502.300

Sensor leaflet

8.0109.8012EN / v11 / 2023-08-15

Metrohm

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Technical Communication
Metrohm AG
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This documentation has been prepared with great care. However, errors can never be entirely ruled out. Please send comments regarding possible errors to the address above.

Disclaimer

Deficiencies arising from circumstances that are not the responsibility of Metrohm, such as improper storage or improper use, etc., are expressly excluded from the warranty. Unauthorized modifications to the product (e.g. conversions or attachments) exclude any liability on the part of the manufacturer for resulting damage and its consequences. Instructions and notes in the Metrohm product documentation must be strictly followed. Otherwise, Metrohm's liability is excluded.

Table of contents

1 Overview

1.1 Combined dCa ISE – Product description

The Combined dCa ISE is a calcium-selective, combined polymer membrane electrode with shockproof membrane for titration, direct measurement and standard addition. The Combined dCa ISE is a dTrode (digital electrode) for OMNIS.

1.2 Combined dCa ISE – Overview

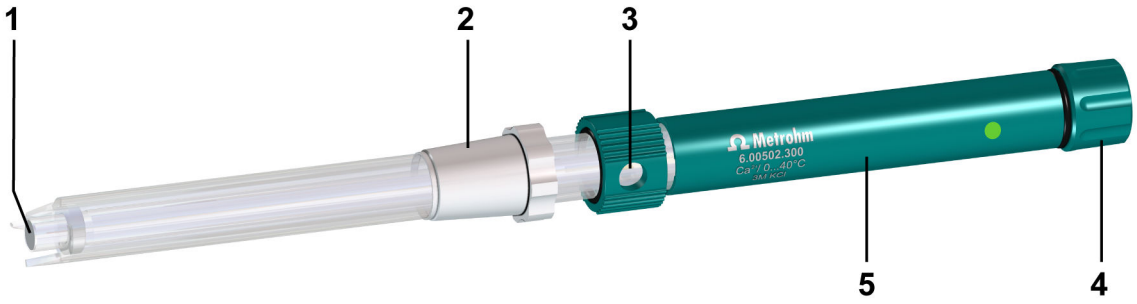


Figure 1 Combined dCa ISE

1	**Sensor surface**	**2**	**Ground-joint sleeve SGJ 14/15, movable**
3	**Filler opening**	**4**	**Protective cap**
5	**Electrode head**		

2 Functional description

2.1 Ion-selective electrode – Functional description

An ion-selective electrode only responds to a specific ion in the solution and, ideally, it does not show any change in potential with other ions present.

The measuring ions of the sample solution reach the membrane surface of the ion-selective electrode, after a certain amount of time an equilibrium is established. An electrochemical potential is created between the measuring solution and the membrane.

3 Delivery and packaging

3.1 Delivery

Inspect the delivery immediately upon receipt:

- Check the delivery against the delivery note to ensure completeness.
- Check the product for damage.
- If the delivery is incomplete or damaged, contact your regional Metrohm representative.

3.2 Packaging

The product and accessories are supplied in protective special packaging. Keep this packaging to ensure safe transportation of the product. If a transport locking device is present, keep this as well for future reuse.

3.3 Unpacking and checking the sensor

Defective sensors must be sent back for warranty processing within two months (starting from the day of delivery).

Required accessories:

- Tool for fixed sensors (included)

1 Unpacking the sensor

Remove the sensor with storage vessel from the packaging.

2 Removing the storage vessel

NOTICE

When applied incorrectly, the sensor could be released too abruptly or sensor parts could become damaged.

This will render the sensor unusable and it will need to be replaced.

Measures to be avoided:

- Avoid applying strong pressure to the tool.
- Observe the direction of movement of the tool.

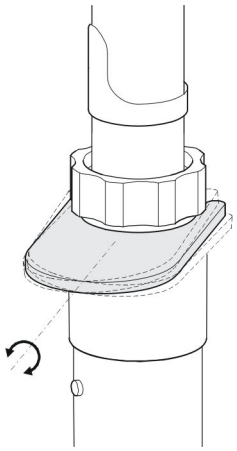


Figure 2 Loosening the sensor from the storage vessel

- Hold the sensor and storage vessel firmly in your hand so that the sensor cannot slip away.
- Position the tool between the storage vessel and the ground-joint sleeve.
- **Carefully** push the tool to the side to release the sensor.
  Do not tip the tool **forwards**!

If the sensor exhibits clear optical defects or damage, then the sensor will already be considered defective and should be sent in. Step 3 is not required in such cases.

3 Checking the sensor for proper function

- **Preparing the sensor:**
  (see "Preparing the Combined dCa ISE", chapter 4.1, page 6)
- **Checking the sensor:**
  (see "Assessing the Combined dCa ISE", chapter 6.3, page 13)

3.4 Storing the Combined dCa ISE

1 For short periods

- Screw the protective cap *(1-**4**)* onto the electrode head *(1-**5**)*.
- Store the electrode in the storage vessel. When doing so, ensure that the sensor surface *(1-**1**)* is immersed in the storage solution.

Use 0.01 mol/L of calcium chloride as the storage solution.

2 For longer periods

- Screw the protective cap *(1-**4**)* onto the electrode head *(1-**5**)*.
- Rinse the electrode and dry the outer shaft of the electrode.

Metrohm recommends maintaining some residual moisture between the inner pipe and the 3 protection flaps to keep the electrode ready for use.

4 Installation

4.1 Preparing the Combined dCa ISE

The ion-selective electrode must be prepared in the following cases:

- Prior to first-time use
- After prolonged pauses
- Between precipitation titrations

NOTICE

Incorrect handling of the electrode

The electrode becomes unusable and must be replaced.

Measures to be avoided:

- Do not touch the sensor surface with your fingers.
- Do not leave the electrode standing in distilled water.
- Do not rub the electrode dry after rinsing.
- Do not bring the electrode into contact with any organic solvents.

Cleaning the electrode

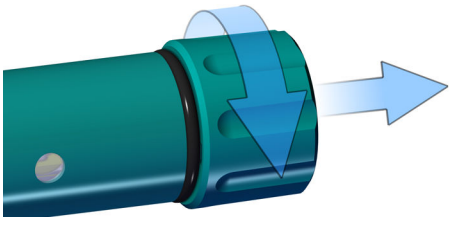
1 Rinse the electrode with distilled water.

The electrode is ready for direct use in most samples and requires no special preliminary treatment.

The electrode is shipped with 3 mol/L of potassium chloride as a reference electrolyte. If the electrode is used for an application with parallel chloride titration, the reference electrolyte should be changed to 1 mol/L of ammonium nitrate.

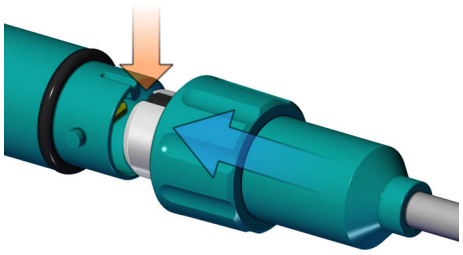
Connecting the electrode

1 Unscrew the protective cap *(1-**4**)*.



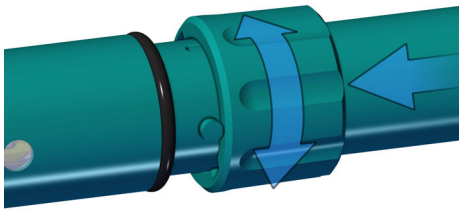
2 Position the cable connection on the electrode head such that the slot in the cable connection is on the guide lug of the electrode head (orange arrow).

Push the socket in the cable connection into the plug inside the electrode head.

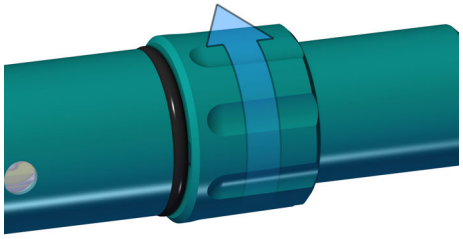


3 Push the outer ring of the cable connection over the electrode head.

Ensure that the guide lugs in the electrode head are in the grooves of the cable connection.



4 Push the cable connection onto the electrode head until it snaps in place.



When an electrode is to be prepared during ongoing operations or if an electrode is to be replaced, then the electrode must be taken out of the titration head and the electrode cable must be removed. To remove the cable, hold the cable connection firmly, do **not** pull on the cable.

4.2 Assembling the sensor

The sensor must be firmly installed on the titration head.

For automatic processes, ensure that the cables have enough room to move.

During the titration, it is important that the solution is mixed well. The stirring rate should be high enough to form a small vortex. If the stirring rate is too high, then air bubbles will be aspirated. These may result in incorrect measured values. If the stirring rate is too low, then the solution is only mixed slowly and the reaction time or titration time increases accordingly.

In order for the measurement to be taken in a well-mixed solution after the addition of the titrant, the titration tip should be positioned where turbulence is high. Furthermore, the distance between the addition of the titrant and the sensor should be as large as possible. Take into account the stirring direction (counterclockwise or clockwise) when positioning the sensor and titration tip.

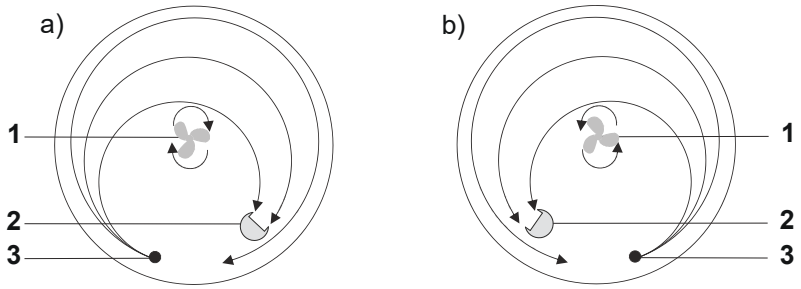


Figure 3 Diagrams showing rod stirrer, sensor and titration tip during a titration. a) clockwise stirring direction, b) counterclockwise stirring direction.

1	**Rod stirrer**	**2**	**Sensor**
3	**Titration tip**		

5 Operation and control

5.1 Combined dCa ISE – Measurement procedures

Titration

Ion-selective electrodes are well-suited for potentiometric titrations. The resulting titration curves are usually S-shaped and can be evaluated well with automatic titrators.

For application advice on working with ion-selective electrodes, please go to *www.metrohm.com*.

Direct measurement with calibration

The ion activity in the sample is interpolated by means of a calibration curve. Establish the calibration curve with standard solutions. The expected ion activity in the sample should lie in the mid-concentration range of the standard solutions.

Since the concentration of an ion is usually to be determined (rather than its ion activity), perform measurements at a fixed ionic strength. The ionic strength is measured in an ISA (Ionic Strength Adjuster) solution or a TISAB (Total Ionic Strength Adjustment Buffer) solution. ISA/TISAB solutions have a high ionic strength so that the various contributions of the measuring ion to the ion strength can be ignored.

For calcium, it is preferable to use a 1 mol/L potassium chloride solution.

Measure samples and calibration standards under identical measuring conditions. The temperature of the standard solutions and the sample solutions should be the same as far as possible, and the temperature should vary as little as possible during measurements.

- In order to guarantee reliable results, periodically execute a control measurement with a calibration standard (e.g. daily).
- Establish a new calibration curve if the deviation is deemed unacceptable.

Standard addition / standard subtraction

In the standard addition method, a defined quantity of the ion to be determined is added to a known volume of the sample (in several increments). Normally, ISA/TISAB solutions are used in this process. The unknown concentration can be calculated from the resulting potential differences between the sample and the sample with added standard solution. This calculation is performed automatically by modern ion meters.

The volume of the added standard solutions should not exceed 25% of the sample volume, and the concentration of the standard solutions should be as high as possible (in order to be able to rule out dilution effects). The potential differences between the increments should be con-

stant and amount to at least 10 mV. Avoid temperature differences between the standard solution and the sample solution. In addition, execute at least 3 additions.

In the standard subtraction method, a solution that eliminates the ion to be determined is added (complexation or precipitation). Apart from that, the same conditions apply as for standard addition. However, this method is rarely used.

6 Maintenance

6.1 Combined dCa ISE – Changing/refilling the electrolyte

1. Open the filler opening *(1-**3**)* by turning it.
2. Use a plastic pipette to empty the electrode.
3. Rinse the inside of the electrode with the new electrolyte.
4. Fill the electrode with electrolyte up to the filler opening.
5. Close the filler opening *(1-**3**)*.

6.2 Cleaning an ion-selective electrode

NOTICE

Damage to the ion-selective electrode caused by ultrasound.

The electrode becomes unusable and must be replaced.

Measures to be avoided:

- **Never** clean ion-selective electrodes in ultrasonic baths.

1. Rinse the electrode with distilled water after each measurement or titration.
2. Check whether the measurement surface of the electrode is clean.

   The surface must be kept clean at all times before the measurement.

6.3 Assessing the Combined dCa ISE

1. Measure $c(Ca^{2+}) = 10^{-4}$ mol/L standard solution and write down the potential.

2. Measure $c(Ca^{2+}) = 10^{-3}$ mol/L standard solution and write down the potential.

3. Calculate the change of potentials with the 2 previously measured potentials:

   The value has to be at least 23.7 mV (at 25 °C) (80% of the theoretical slope).

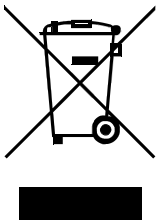
   If the value cannot be reached, replace the electrode.

7 Combined dCa ISE – Troubleshooting

If additional or other types of faults occur, ensure the following points:

- Is the electrode cable screwed on and plugged in correctly?
- Is the electrode cable functional?
- Is the sensor surface clean and intact?
- Is the electrode new? Information on service life: *(see "Service life of the dCa ISE with polymer membrane", chapter 10.2, page 19)*

8 Electrode – Disposal



Properly dispose of chemicals and of the product to reduce negative effects on the environment and public health.

Proceed as follows to dispose of the electrode:

1. Use a plastic pipette to remove the electrolyte from the electrode.
2. Dispose of the electrolyte in accordance with the legal provisions.
3. Put the electrode in electronic waste recycling.

Local authorities, waste disposal companies or dealers provide more detailed information on disposal. Observe the WEEE EU directive (WEEE = Waste Electrical and Electronic Equipment) for the proper disposal of waste electronic equipment within the European Union.

9 Technical specifications

9.1 Ambient conditions

Nominal function range	+5 to +45 °C	at max. 80% relative humidity, non-condensing
Storage	+5 to +45 °C	

9.2 Combined dCa ISE – Dimensions

Measurements

Shaft diameter	12 mm
Maximum installation length	113 mm

9.3 Combined dCa ISE – Housing

Materials

Shaft material	PMMA	poly(methyl methacrylate)
	PP	polypropylene
Inner pipe	PMMA	poly(methyl methacrylate)

9.4 Ion-selective electrode – Connectors specifications

Connector		Metrohm plug-in head Q

9.5 dTrodes – Display specifications

Status display	LED	green-red

9.6 Combined dCa ISE – Measurement specifications

pH range	0–12	
Temperature range	0–40 °C	
Measuring range		
Ion concentration	$5*10^{-7}$–1 mol/L	
Minimum immersion depth	10 mm	

9.7 dTrode – Analog measurement connection

Potentiometric		
Measuring range	–1,900 to +1,900 mV	
Resolution	1.28 µV	
Measuring accuracy	±0.5 mV	in the measuring range –1,900 mV to +1,900 mV
Input resistance	≥ $1*10^{12}$ Ω	
Offset current	≤ ±$1*10^{-12}$ A	
Temperature		
Pt1000		
Measuring range	–150 to +250 °C	
Resolution	approx. 0.002 °C	

Measuring accuracy	±0.4 °C	in the measuring range –20.0 to +150.0 °C

Reference conditions

Relative humidity	≤60%	
Ambient temperature	+25 °C (±3 °C)	
Instrument status		min. 30 minutes in operation

Measuring accuracy

applies for all measuring ranges without sensor error, under reference conditions, measuring interval 100 ms

Valid for the measurement contacts of the analog measurement connection installed in the sensor. These connections are not accessible after installation.

10 Appendix

10.1 Additional information

ISA/TISAB solutions

Table 1 ISA/TISAB solutions

Measuring ion	ISA/TISAB	For 100 mL of solution	Remarks
Ca^{2+}	KCl 1 mol/L	7.46 g	

Interfering ions

The concentrations in mol/L of the interfering ions, which generate an analysis error of approximately 10%, are specified in the following table.

Table 2 Interfering ions

Measuring ion	Interferences
Ca^{2+}	$c(Na^{+}) < 0.24$; $c(K^{+}) < 0.4$; $c(Mg^{2+}) < 18$; $c(H^{+}) < 0.12$; $c(OH^{-}) < 0.11$; $c(Cu^{2+}) < 8 \cdot 10^{-2}$; $c(Pb^{2+}) < 3.5 \cdot 10^{-2}$; $c(Zn^{2+}) < 0.22$; $c(Fe^{2+}) < 0.45$

10.2 Service life of the dCa ISE with polymer membrane

The service life of the electrode is limited. The average service life in regular laboratory use is approx. half a year. This value greatly depends on the application used and on the maintenance of the electrode.

The service life is also shortened if the electrode is not used, i.e. only kept in storage.

Measures to reduce the decrease in service life:

- Do not purchase stocks of electrodes for future use.
- Do not store electrodes unused for long periods.
- Guidelines for storage: *page 16*
- Instructions for storage: *(see "Storing the Combined dCa ISE", chapter 3.4, page 4)*

Metrohm recommends performing regular checks of stored electrodes: *(see "Assessing the Combined dCa ISE", chapter 6.3, page 13)*