

OMNIS Coulometer



2.1018.0xx0

Product manual

8.1018.8002EN / 2025-05-16



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Technical Communication
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CH-9100 Herisau

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1 Overview

1.1 OMNIS Coulometer – Product description


The OMNIS Coulometer is the central instrument of an OMNIS titration system for coulometric Karl Fischer titrations. Coulometric water content determination titrations according to Karl Fischer can be carried out with the aid of corresponding accessories.

1.2 OMNIS Coulometer – Product versions

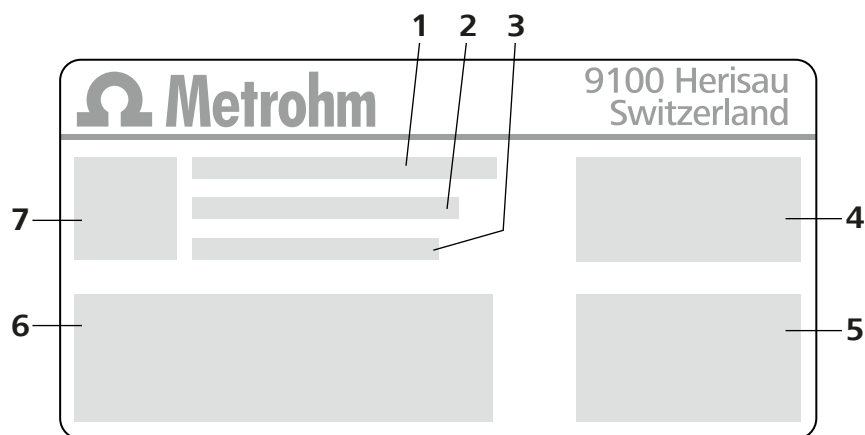
The product is available in the following versions:

Table 1 Product versions

Article number	Designation	Version feature
2.1018.0010	OMNIS Coulometer	without magnetic stirrer
2.1018.0020	OMNIS Coulometer	with integrated magnetic stirrer

 Information on function licenses is available on the [Metrohm website](#) or from the regional Metrohm representative.

The article number and serial number for identification of the product can be found on the type label:



1 (01) = Article number in accordance with GS1 standard

2 (21) = Serial number

3 (240) = Metrohm article number

4 Certification




5 Technical specifications

6 Certification

7 QR code

1.3 About the documentation

Possible depictions in the documentation:

Depiction	Meaning
(5-12)	Cross-reference to figure legend (Figure number - <i>Element in the figure</i>)
1	Instruction step
Method	Parameters, menu items, tabs, and dialogs
File ▶ New	Menu path
[Continue]	Button or key
	Supplementary information to the descriptive text
	Note In graphics, orange arrows or frames indicate the reference to the descriptive text. The relevant elements may also be colored orange.
	Movement In graphics, blue arrows indicate the movement direction. The elements to be moved may also be colored blue.

1.4 Further information


Additional information on the OMNIS Coulometer is available on the following pages:

- Metrohm website <https://www.metrohm.com> – Overview of product family, document as PDF, details of the accessories, and information on applications.
- Software help of the OMNIS Software <https://guide.metrohm.com> – Thematically filtered individual content, videos, information on control software.

1.5 Displaying the accessories

Up-to-date information on the scope of delivery and on optional accessories can be found on the Metrohm website.

1 Searching for a product on the website


- Go to <https://www.metrohm.com>.
- Click on .
- Enter the article number of the product into the search field and press **[Enter]**.
 - Article number: See *OMNIS Coulometer – Product versions, chapter 1.2, page 1*
- In the result list, click on the desired product.


Detailed information regarding the product is displayed.

2 Displaying the accessories

- Scroll down (accessories subject to availability):
 - Included parts
 - Optional parts

3 Downloading the accessories list (included and optional parts)

- Click on  to download the accessories list as a PDF.

 Metrohm recommends keeping the downloaded PDF for reference purposes.

2.2 Responsibility of the operator

The operator must ensure that basic regulations on occupational safety and accident prevention in chemical laboratories are observed. The operator has the following responsibilities:

- Instruct personnel in the safe handling of the product.
- Train personnel in the use of the product according to the user documentation (e.g. install, operate, clean, eliminate faults).
- Train staff on basic occupational safety and accident prevention regulations.
- Provide personal protective equipment (e.g. protective glasses, gloves).
- Provide suitable tools and equipment to carry out the work safely.

The product may be used only when it is in perfect condition. The following measures are required to ensure the safe operation of the product:

- Check the condition of the product before use.
- Remedy defects and malfunctions immediately.
- Maintain and clean the product regularly.

2.3 Requirements for operating personnel

Only qualified personnel may operate the product. Qualified personnel are persons who meet the following requirements:

- Basic regulations on occupational safety and accident prevention for chemical laboratories are known and complied with.
- Knowledge of handling hazardous chemicals is present. Personnel have the ability to recognize and avoid potential dangers.
- Knowledge regarding the application of fire prevention measures for laboratories is available.
- Safety-relevant information is communicated and understood. The personnel can operate the product safely.
- The user documentation has been read and understood. The personnel operate the product according to the instructions in the user documentation.

2.4.3 **Danger from highly flammable substances**

Using highly flammable substances or gases may cause fires or explosions. To avoid danger from highly flammable substances, observe the following:

- Avoid ignition sources.
- Use protective grounding.
- Use exhaust equipment.

2.4.4 **Danger from leaking liquids**












Leaking liquids may cause injuries and may damage the product. To avoid danger from leaking liquids, observe the following:

- Check the product and its accessories for leakages and loose connections.
- Replace leaking components and connecting elements without delay.
- Tighten loose connecting elements.
- Do not loosen tubing connections under pressure.
- Do not remove aspiration tubing under pressure.
- Carefully pull the tubing ends out of the vessels.
- Carefully allow liquids from the tubing to drain into suitable vessels.
- Insert the tubing tips completely into the vessels.
- Remove and dispose of escaping liquids in accordance with regulations.
- If you suspect that liquid has penetrated the instrument, disconnect the instrument from the energy supply. Then have the instrument checked by a regional Metrohm service representative.

2.4.5 **Danger during transport of the product**

Chemical or biological substances may be spilled during the transport of the product. Parts of the product may fall down or may be damaged. There is a risk of injury from chemical or biological substances and pieces of broken glass. To ensure safe transport, observe the following:

- Remove loose parts (e.g. sample racks, sample vessels, bottles) before transport.
- Remove liquids.
- Lift and transport the product with both hands on the base plate.
- Lift and transport heavy products only according to instructions.

Warning signs / meaning	Warning signs / meaning
 General warning sign	 Warning of hot surface
 Warning of sharp object (cut/puncture)	 Warning of hand injuries (crushing)
 Warning of electrical voltage	 Warning of corrosive substances
 Warning of optical radiation	 Warning of a laser beam
 Warning of flammable materials	 Warning of biological hazard
 Warning of toxic materials	



3 Functional description

3.1 OMNIS Coulometer – Overview

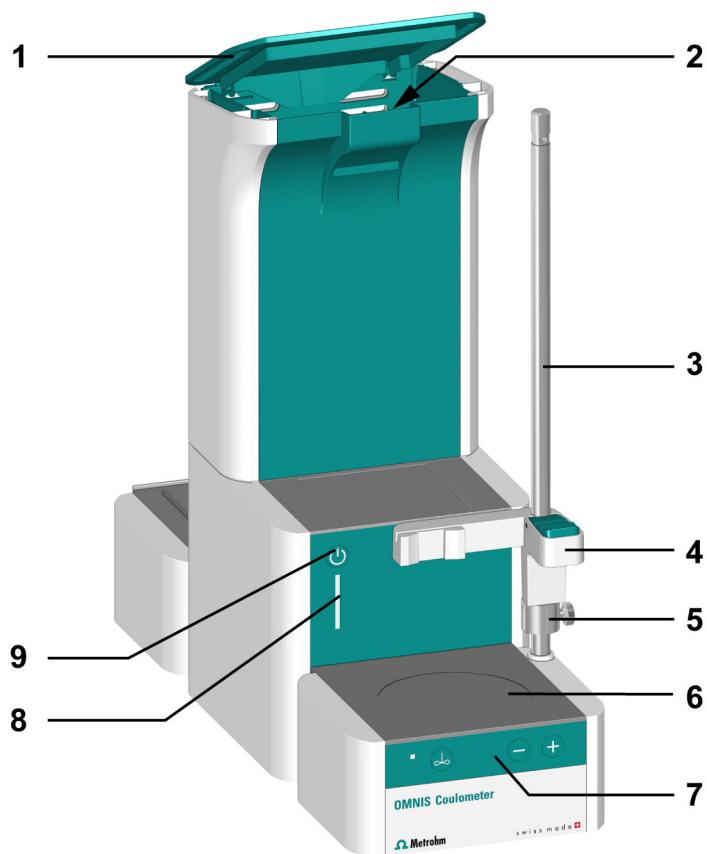


Figure 1 OMNIS Coulometer (with magnetic stirrer) – Overview

1	Lid	2	Internal measuring interface
3	Support rod (6.2016.050)	4	Titration cell holder (6.02047.000)
5	Clamping ring (6.2013.010)	6	Magnetic stirrer
7	Control bar of the magnetic stirrer	8	Status display
9	On/off switch		

3.1.1 Coulometric Karl Fischer titration cell – Versions

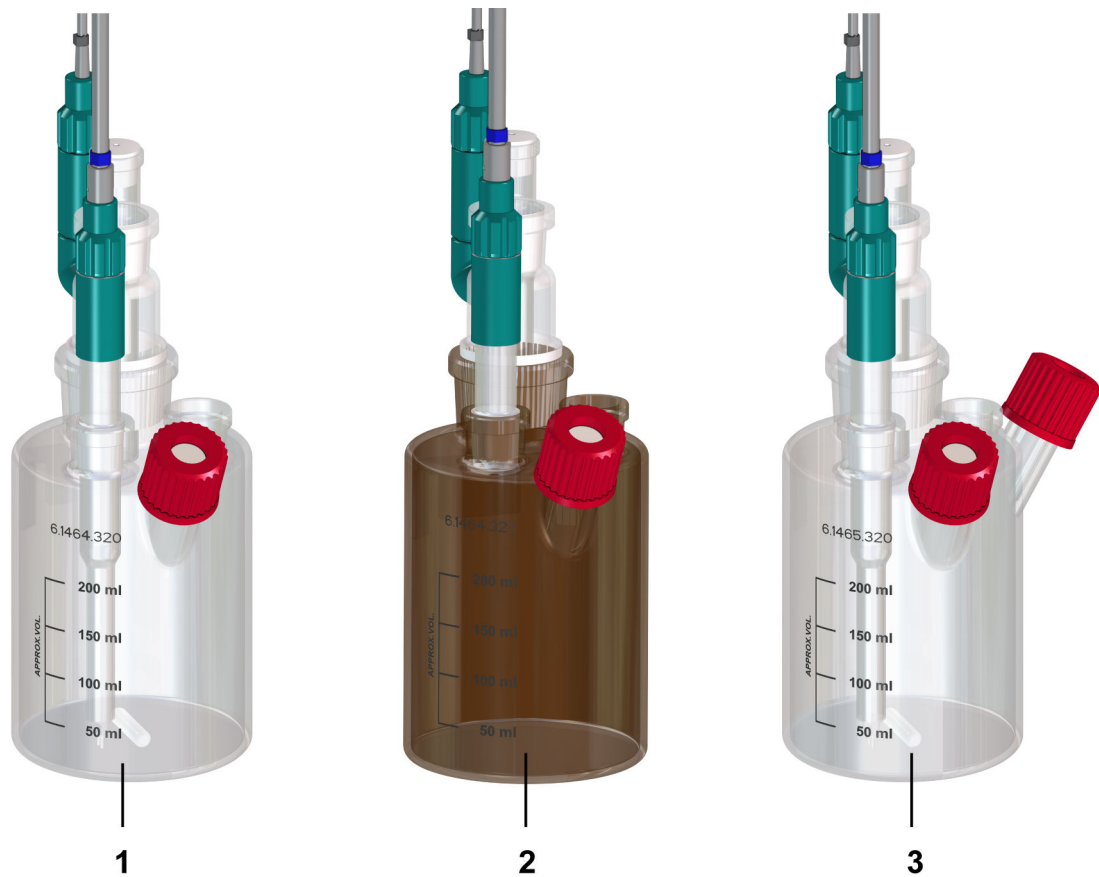


Figure 2 3 versions of the coulometric Karl Fischer titration cell

- | | |
|---------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------|
| <p>1 Karl Fischer titration cell / 80–250 mL / coulometric (6.1464.320)</p> | <p>2 Karl Fischer titration cell made of amber glass / 80–250 mL / coulometric (6.1464.323)</p> |
| <p>3 Karl Fischer titration cell with 2 side openings / 80–250 mL / coulometric (6.1465.320)</p> | |



3.1.2 Coulometric Karl Fischer titration cell – Overview

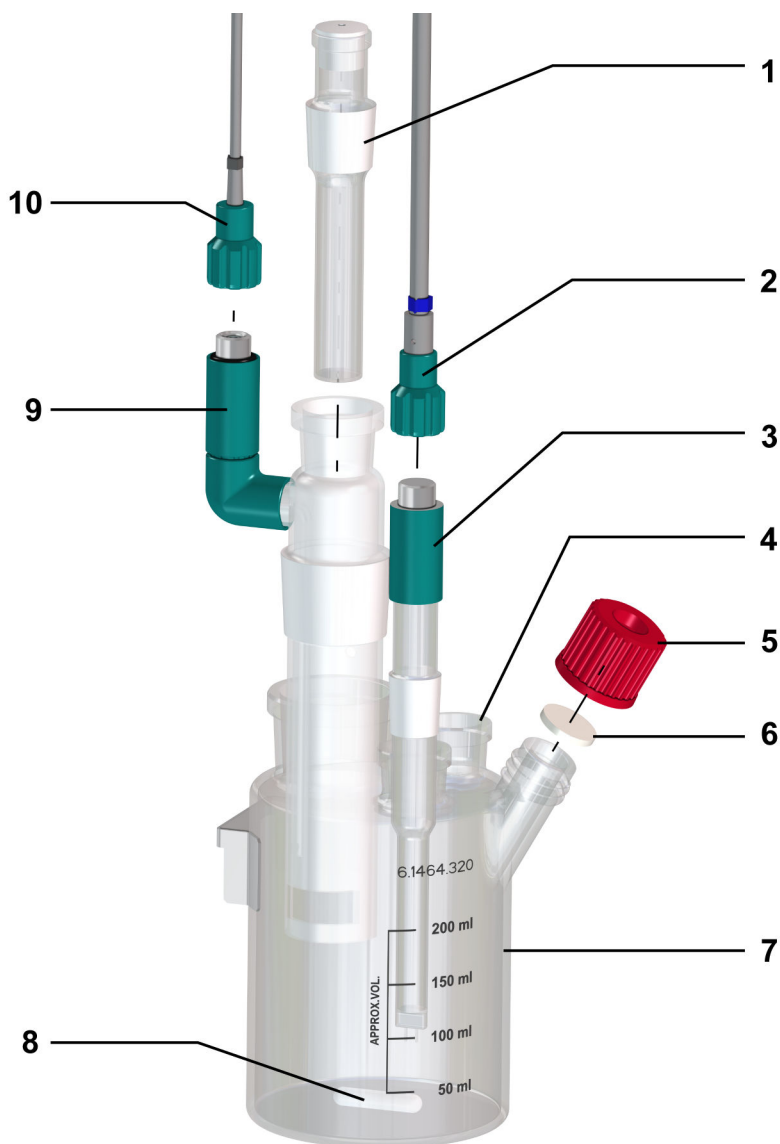


Figure 3 Coulometric Karl Fischer titration cell (equipped) – Overview

- | | |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------|
| <p>1 Adsorber tube (6.1403.030)
 With ground-joint sleeve (6.2713.020)
 In combination with an OMNIS Sample Robot Oven, if necessary with tubing olive (6.1808.310) for a tubing for discharging gases.</p> | <p>2 Indicator electrode cable (6.02104.040)
 With blue coding for polarizable metal electrodes</p> |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------|

3 Indicator electrode With ground-joint sleeve (6.2713.000) e.g. double Pt wire electrode for coulometry (6.0341.100)	4 Opening for application versions Ground-joint stopper (6.1437.000) with ground-joint sleeve (6.2713.000)
5 Screw cap (6.2701.040)	6 Septum (6.1448.020) for manual sample addition
7 Karl Fischer titration cell / 80–250 mL / coulometric with attached metal clip for titration cell holder	8 Stirring bar 25 mm (6.1903.030)
9 Generator electrode With ground-joint sleeve (6.2713.010) Without diaphragm (6.00349.100) or with diaphragm (6.00348.100)	10 Generator electrode cable (6.2104.620) With gray coding for generator electrodes


3.2 OMNIS Coulometer – Functional description

The OMNIS Coulometer consists of the following functional units:

- Connectors to the power grid and the Ethernet network
- Interfaces for connecting additional modules
- Connector for 1 generator electrode
- Measuring input **INPUT 1** for 1 temperature sensor / 1 pH electrode / 1 polarizable metal electrode (observe color coding)
- Measuring input **INPUT 2** for 1 temperature sensor / 1 pH electrode (observe color coding)
- 1 optional integrated magnetic stirrer, depending on the product version

The OMNIS Coulometer contains the logic necessary for controlling the titration system. The OMNIS Coulometer is connected to the energy supply and the Ethernet network. All of the other modules of the titration system are connected with the OMNIS Coulometer.

The OMNIS Coulometer connected to the Ethernet network can be operated with the OMNIS Software. The OMNIS Coulometer is then responsible for the energy supply of all modules of the titration system as well, as for the communication of the titration system with the OMNIS Software.

 The range of functions of the OMNIS Coulometer is defined in the selected function license.

See also

OMNIS Coulometer – Interfaces (chapter 3.5, page 16)



3.2.1 Magnetic stirrer – Functional description

The magnetic stirrer ensures that the sample is well mixed. The stirring rate can be adjusted depending on the amount and viscosity of the sample. The magnetic stirrer is operated via the instrument control bar or via the OMNIS Software.

3.2.2 Coulometric Karl Fischer titration cell – Functional description

The coulometric Karl Fischer titration cell is a closed vessel for the water content determination according to Karl Fischer. The equipping differs, depending on the application version and application. There are 3 versions of the coulometric Karl Fischer titration cell for fulfilling different applications:

- Coulometric Karl Fischer titration cell (6.1464.320),
- Coulometric Karl Fischer titration cell (6.1464.323) made of amber glass,
- Coulometric Karl Fischer titration cell (6.1465.320) with 2 side openings.

The coulometric Karl Fischer titration cell is attached to a titration cell holder on the support rod. The amber glass version is recommended for light-sensitive materials.

3.3 OMNIS main instrument – Indicators and controls

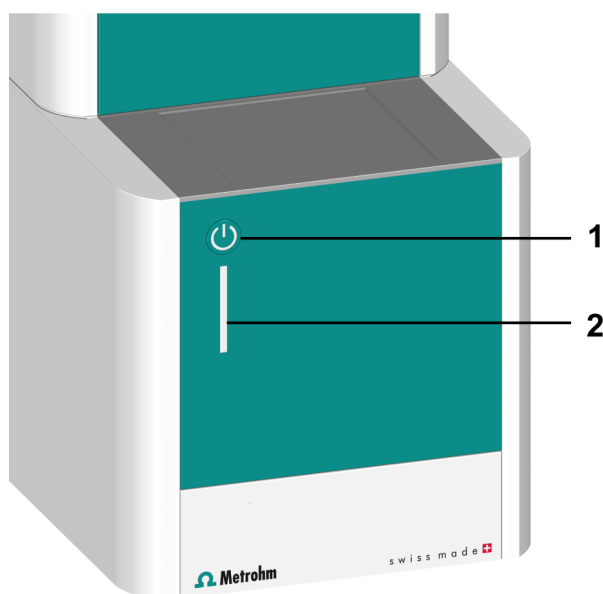


Figure 4 OMNIS main instrument – Indicators and controls

1 On/off switch

2 Status display
multi-colored

Indicators

The status of the OMNIS main instrument is displayed with the status display (4-2) using different colors (see *System – Signals, page 15*).

Controls







The on/off switch (4-1) is used for the hardware-side operation of the OMNIS main instrument.

Table 3 Behavior of the on/off switch

Pressure duration	Acoustic signal	Function
Short pressing (1–5 s)	Beep sound after 1 s	Switch on the instrument. Shut down the instrument.
Very long pressing (> 10 s)	Continuous beep sound after 8 s	Force a shutdown.

3.4 System – Signals

System components with status indicators show their operating status with colors and/or flashing patterns. The meaning of the colors and flashing patterns is explained in the following table.

Visual signal		Meaning
	LED lights up yellow.	System start or initialization
	LED flashes yellow (slowly).	Ready for connection setup or locking
	LED flashes yellow (fast).	Connection setup started or locking underway
	LED lights up green.	Ready for operation
	LED flashes green (slowly).	In operation
	LED flashes red (fast).	Malfunction or error

Some system components only use part of the explained flashing patterns.



3.5 OMNIS Coulometer – Interfaces

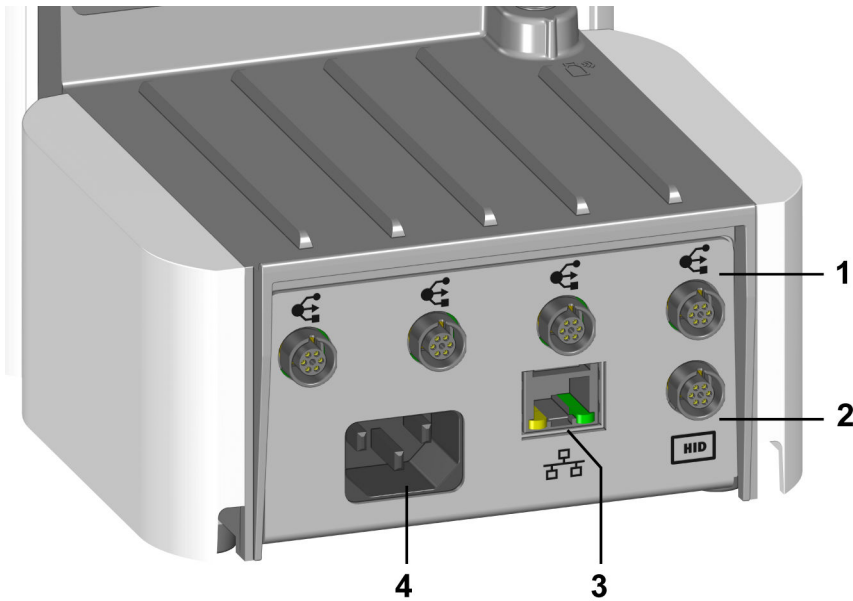


Figure 5 OMNIS Coulometer – Interfaces and connectors

1 MDL connectors

MDL = Metrohm Device Link.
 Connection socket for connecting cables between OMNIS products

2 HID connector

HID = Human Interactive Device.
 Connection socket for external operating units

3 Ethernet network connection or LAN connector

LAN = Local Area Network.
 Connection socket for a connecting cable to the local network

4 Power socket

Connection socket for the energy supply

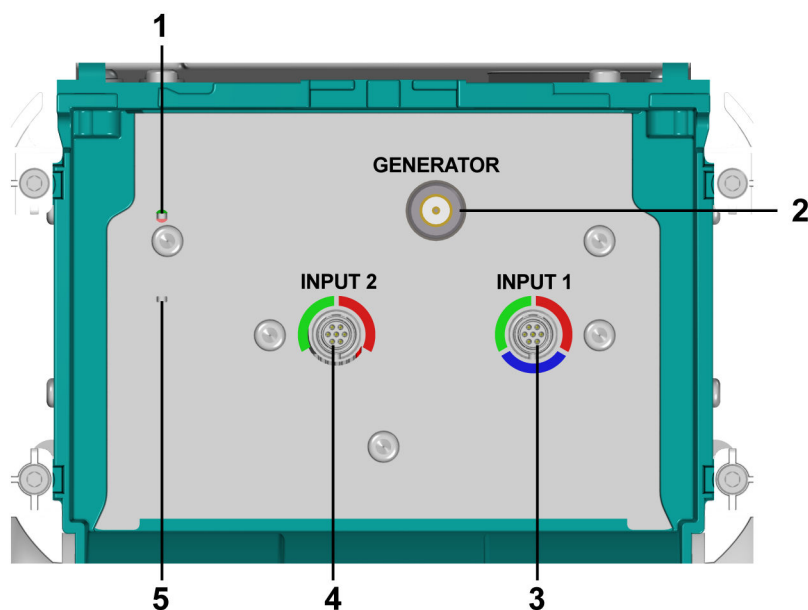


Figure 6 OMNIS Coulometer or OMNIS Coulometer Module – Internal measuring interface

<p>1 Indicator Indicator for the GENERATOR outlet</p>	<p>2 GENERATOR Connector for generator electrode (gray coding)</p>
<p>3 INPUT 1 Temperature sensor (red coding) or Measuring input for polarizable metal electrode (blue coding) or Measuring input for potentiometric sensor (green coding)</p>	<p>4 INPUT 2 Temperature sensor (red coding) or Measuring input for potentiometric sensor (green coding)</p>
<p>5 Indicator Indicator for the internal measuring interface</p>	

Measuring inputs INPUT 1 and INPUT 2

The measuring inputs **INPUT 1** and **INPUT 2** are marked with colored circle segments. The markings indicate that only certain types of electrode cables may be plugged into the connection socket:

Table 4 Meaning of the colors

Red	The connector supports temperature sensors.
Blue	The connector supports polarizable metal electrodes.
Green	The connector supports potentiometric sensors.
Gray	The connector supports a generator electrode.



4 Delivery and transport

4.1 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.

4.2 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.

5 Installation

5.1 Installation by Metrohm

Installation and start-up of the system is always carried out by the regional Metrohm service representative.

5.2 Setup location

The product is only suitable for operation indoors and may not be used in explosive environments.

The following requirements apply to the setup location:

- The room is well-ventilated and protected against both direct sunlight and excessive temperature fluctuations.
- The setup space is stable and free of vibrations. The setup space must be suitable for the dimensions and weight of the components (see technical specifications).
- All cables and connectors are accessible during operation. The cables are safely installed (no tripping hazards).
- The workplace is ergonomically designed and ensures trouble-free operation of the product.

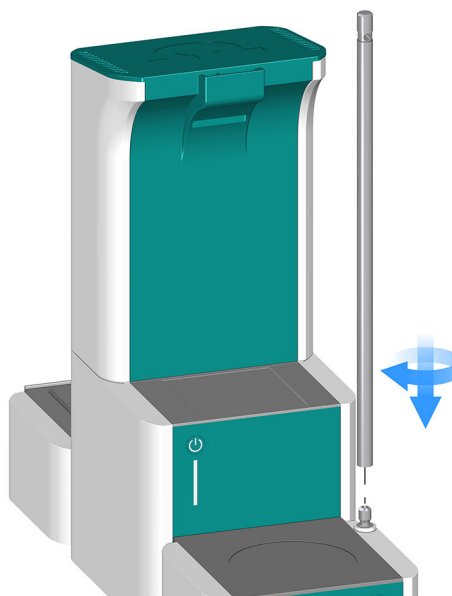
5.3 Mounting the magnetic stirrer accessories

Required accessories:

- Support rod (6.2016.050)
- Clamping ring (6.2013.010)
- Titration cell holder (6.02047.000)

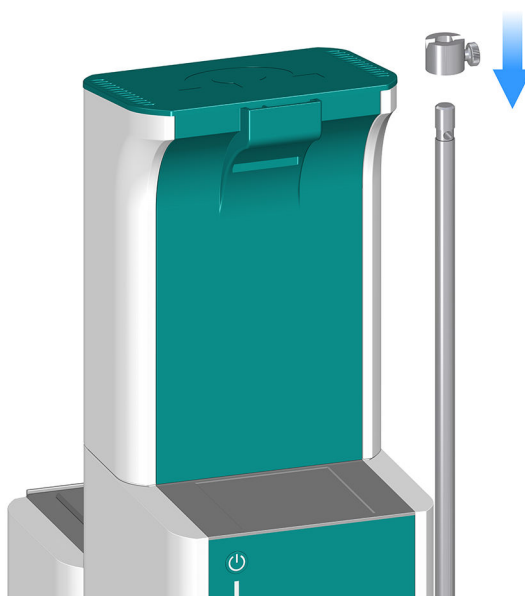


1 Mounting the support rod



- Screw the support rod onto the stand attachment.

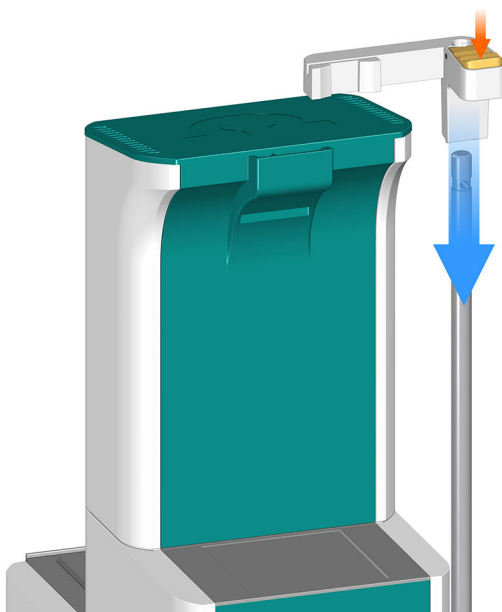
2 Mounting the clamping ring



- Push the clamping ring over the support rod with the groove facing upward.



3 Mounting the titration cell holder



- Press the green locking lever on the titration cell holder.
- Push the titration cell holder over the support rod.
- To fix in place, release the green locking lever at the desired height.

5.4 Replacing the adsorber material





Depending on the OMNIS product, different adsorber cartridges or adsorber tubes are available.

Table 5 Available adsorber cartridges or adsorber tubes

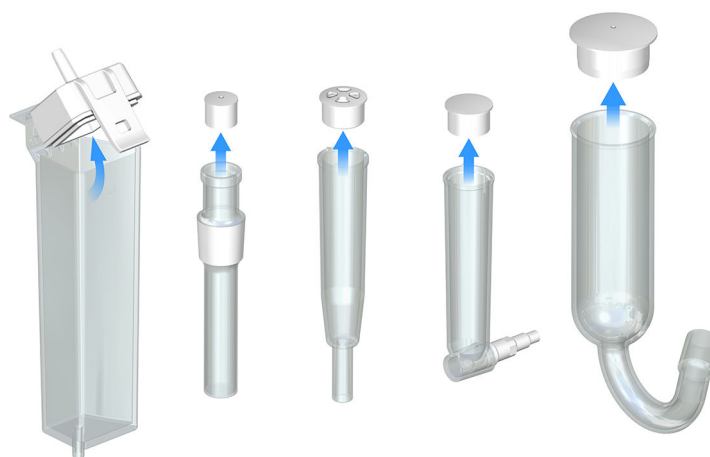
Adsorber cartridge / adsorber tube	Article number	Figure
Adsorber cartridge for OMNIS Solvent Module	6.01807.000	





Adsorber cartridge / adsorber tube	Article number	Figure
Adsorber tube for coulometric Karl Fischer titration cell	6.1403.030	
Adsorber tube for volumetric Karl Fischer titration cell	6.01406.010	
Adsorber tube for a cylinder unit OMNIS	6.1619.020	
Adsorber tube for waste bottle for OMNIS Dosing Module	6.1609.000	

1 Removing the lid from the housing



- Adsorber cartridge: Unlatch and remove the lid including the seal from the housing.
- Adsorber tube: Remove the lid by pulling it out of the housing.

2 Removing the molecular sieve (if present)

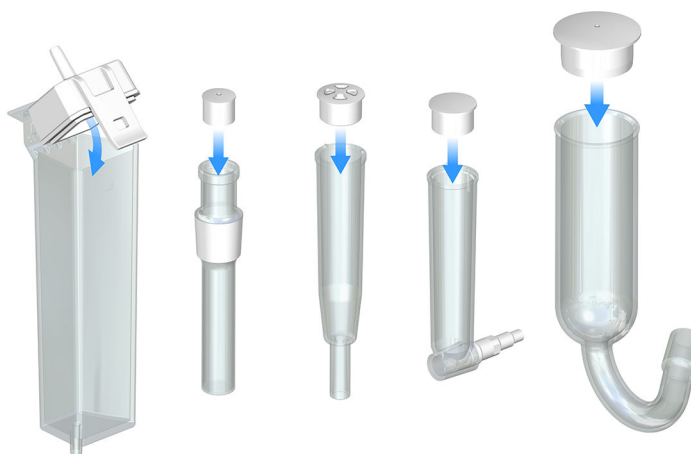
- Remove the molecular sieve and regenerate for at least 24 hours in the drying oven at 300 °C. Place in a desiccator to cool down and then seal airtight in a glass bottle, see also [FAQ on Karl Fischer titration](#).

3 Filling the molecular sieve

- Adsorber cartridge: Place a base-covering cotton plug loosely into the housing at the bottom. Do not pack the wad of cotton too tightly as sufficient gas flow must be possible. Use a molecular sieve to fill the housing to approx. 1 cm under the housing edge.
- Adsorber tube: Place a small cotton plug on the molecular sieve. Do not pack the wad of cotton too tightly as sufficient gas flow must be possible.

4 Sealing the housing with the lid

- **i** Make sure that the sealing surface between the housing and the lid is clean and dry, and that there no residual filling material whatsoever is present.



- Adsorber cartridge: Hook the lid including the seal into the housing side and close it by clicking it into place.
- Adsorber tube: Seal the housing with the lid.

i At moderate humidity, replace the molecular sieve approx. every 6 weeks.

An increase in drift is an indication that the molecular sieve is saturated and that the humidity is therefore entering the Karl Fischer titration cell.

Hint:

After replacing the molecular sieve, write the date on the adsorber housing.

5.5 Equipping the coulometric Karl Fischer titration cell

⚠ CAUTION

Risk of cuts from sharp edges

Cut injuries due to damaged glass parts and pieces of broken glass.

- Handle glass parts (e.g. electrodes, bottles) with care.
- Only use undamaged glass parts.
- Dispose of damaged glass parts immediately.

Preparing the coulometric Karl Fischer titration cell

Prerequisite:

- The adsorber tube of the generator electrode (6.1403.030) is filled with cotton and molecular sieve (see *"Replacing the adsorber material"*, chapter 5.4, page 21).
- When using a dosing module: The adsorber tube for reagent replacement (6.1619.020) is filled with cotton and molecular sieve.


Required accessories:

- Indicator electrode, generator electrode, adsorber tube, etc. (see *"Coulometric Karl Fischer titration cell – Overview"*, chapter 3.1.2, page 12)

- 1 Place a stirring bar in the Karl Fischer titration cell.
- 2 Cut the ground-joint sleeve to the correct length. Ensure that there are no fringes left over.
- 3 Slide the ground-joint sleeves over the ground joints of the electrodes and the adsorber tube. In addition, slide a ground-joint sleeve onto the adapter for application versions.

Equipping the coulometric Karl Fischer titration cell

Prerequisite:

- The Karl Fischer titration cell is prepared.
- 1 Insert the adsorber tube filled with molecular sieve into the generator electrode.
 - 2 Insert the generator electrode into the large ground-joint opening at the rear.
 - 3 Insert the indicator electrode into the left ground-joint opening.
 - 4 Screw an electrode cable with blue coding tightly on the indicator electrode.
 - 5 Screw an electrode cable with gray coding tightly on the generator electrode.
 - 6 Place the septum on the front opening of the titration cell and screw it shut with the screw cap.
 -  Tighten the screw cap only enough so that everything seals. The septum must not bend.

- 7 Fill the Karl Fischer titration cell. (see "Filling the coulometric Karl Fischer titration cell", chapter 5.6, page 26).
- 8 Depending on the application, insert the required adapter into the opening for application versions.

See also

Coulometric Karl Fischer titration cell – Overview (chapter 3.1.2, page 12)

5.6 Filling the coulometric Karl Fischer titration cell

**WARNING****Contact with chemicals**

Chemicals may cause chemical burns.

- Wear personal protective equipment (e.g. protective glasses, gloves).
- Use exhaust equipment when working with vaporizing hazardous substances.

Use of a generator electrode with diaphragm**Prerequisite:**

- The Karl Fischer titration cell is completely equipped with a generator electrode with diaphragm. Pre-dry the components in the oven at 50 °C.

- 1 Remove the adsorber tube from the generator electrode.
- 2 Fill approximately 5 mL of catholyte into the generator electrode.
- 3 Insert the adsorber tube into the generator electrode.
- 4 Remove the right ground-joint stopper from the Karl Fischer titration cell.
- 5 Using a funnel, fill as much analyte into the Karl Fischer titration cell until the 100 mL marking on the titration cell is reached.

The level of the analyte should be roughly 1 to 2 mm above the level of the catholyte.
- 6 Close the ground-joint opening on the right with the ground-joint stopper (with ground-joint sleeve attached).

Use of a generator electrode without diaphragm

Prerequisite:

- The Karl Fischer titration cell is completely equipped with a generator electrode without diaphragm. Pre-dry the components in the oven at 50 °C.

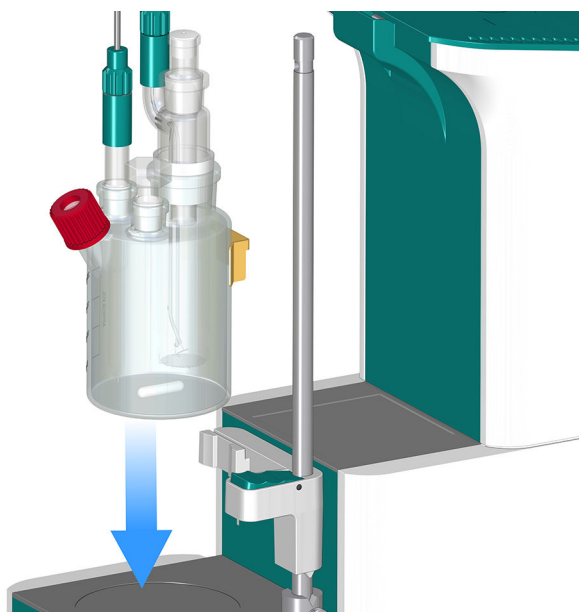
- 1** Remove the right ground-joint stopper from the Karl Fischer titration cell.
- 2** Use a funnel to fill approximately 100 mL of reagent into the Karl Fischer titration cell.
- 3** Close the ground-joint opening on the right with the ground-joint stopper (with ground-joint sleeve attached).

5.7 Mounting the coulometric Karl Fischer titration cell

Prerequisite:

- The support rod is mounted with clamping ring and titration cell holder (see *Mounting the magnetic stirrer accessories*, page 19).

1 Placing the titration cell



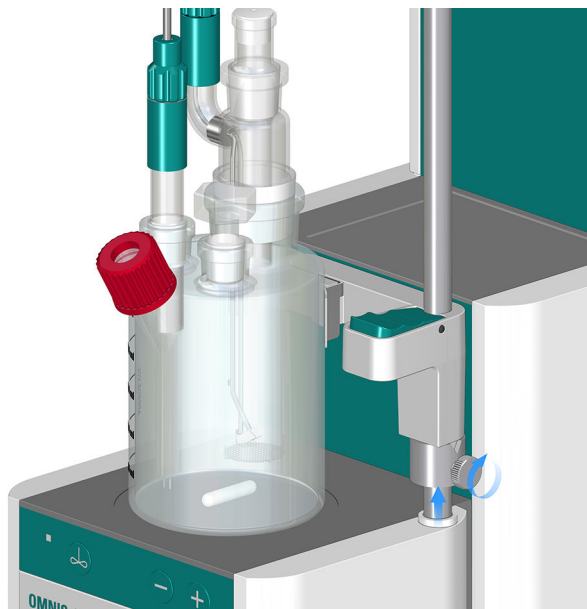
- Slide the metal clip of the coulometric Karl Fischer titration cell over the titration cell holder.



2 Checking the position of the titration cell

- The titration cell is positioned in the center of the magnetic stirrer.

3 Setting the clamping ring



- Push the clamping ring under the titration cell holder.
- Rotate the clamping ring in such a way that the wedge on the titration cell holder fits into the groove of the clamping ring.
- Fix the clamping ring with the knurled screw in position at the desired height.

The clamping ring is used as the lower stop for the titration cell holder. The stop facilitates the correct positioning of the titration cell on the magnetic stirrer.


5.8 Connecting the electrodes

CAUTION

Damage to the indicator electrode

The indicator electrode will be damaged if it is connected to the connector for the generator electrode. The two electrodes have the same plug-in head and can be connected incorrectly. The connectors on the instrument are different.

- Observe the color coding of the electrode cables and the name of the connection sockets:
 - Plug the electrode cable with **gray coding** in the **GENERATOR** connector and mount the generator electrode.
 - Plug the electrode cable with **blue coding** in the **INPUT 1** measuring input and mount the indicator electrode.

-  If the plug cannot be inserted easily, rotate the plug slightly to the right or left using light pressure until it latches in the socket.
 - Align the red dot on the plug with the groove on the measuring input.
 - Plug in the plug until you can feel it snap in.

Connecting the generator electrode and indicator electrode

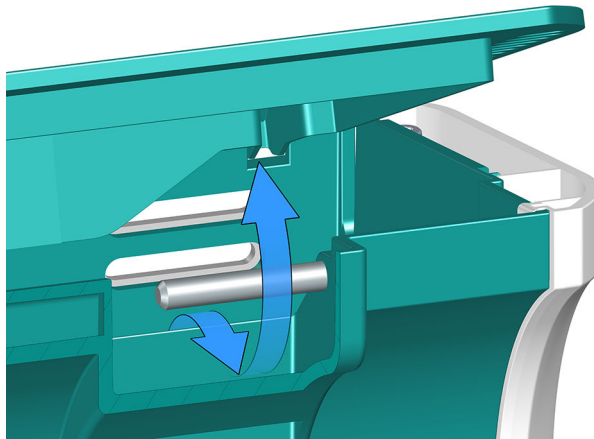
Prerequisite:

- The electrode cable with gray coding is screwed tightly on the generator electrode.
- The electrode cable with blue coding is screwed tightly on the indicator electrode.

- 1** Open the lid on the OMNIS Coulometer or OMNIS Coulometer Module.
- 2** Plug the electrode cable with **gray coding** in the **GENERATOR** connector.
- 3** Plug the electrode cable with **blue coding** in the **INPUT 1** measuring input.



4 Guiding out the cable



Guide the cables out going under the bar.

5 Close the lid.

5.9 Plugging in the power cord



WARNING

Electric shock from electrical potential

Risk of injury by touching live components or through moisture on live parts.

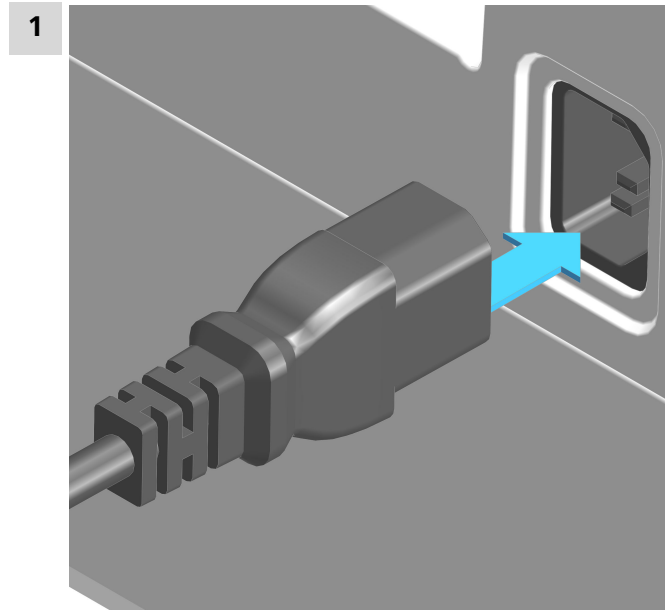
- Never open the housing of the instrument while the power cord is still connected.
- Protect live parts (e.g. power supply unit, power cord, connection sockets) against moisture.
- Unplug the power plug immediately if you suspect that moisture has gotten inside the instrument.
- Only personnel who have been issued Metrohm qualifications may perform service and repair work on electrical and electronic parts.

Required accessories:

- Power cord:
 - Length: max. 2 m
 - Number of conductors: 3, with protective ground
 - Conductor cross-section: 3x min. 0.75 mm² / 18 AWG
- Instrument plug:
 - IEC 60320, type C13, 10 A



- Power plug:
 - 6.2122.XX0 (according to customer requirement), min. 10 A



- Plug the power cord into the product's power socket. Use only permitted power cords.
- Connect the power cord to the energy supply.
- To power down the product, unplug the power cord from the energy supply.



6 Start-up

6.1 Initial start-up by Metrohm

As a basic rule, the initial start-up of the system is carried out by the regional Metrohm service representative.

7 Coulometric titration

The **coulometric Karl Fischer titration** is a variation of the classic water content determination method according to Karl Fischer.

7.1 OMNIS Coulometer – Principle of coulometry according to Karl Fischer

The **coulometric Karl Fischer titration** is a variation of the classic water content determination method according to Karl Fischer. The conventional method works with a methanolic solution of iodine, sulfur dioxide and a base as buffer substance. If an aqueous sample is titrated, then several reactions take place that can be summarized in the following sum equation:



According to the equation above, the I_2 reacts quantitatively with H_2O . This chemical equation serves as a basis for the water content determination.

With the **coulometric Karl Fischer titration**, the necessary iodine is directly and electrochemically generated in the electrolyte containing iodine. Between the amount of electric charge and the amount of generated iodine, there is a strictly quantitative relationship, which is used for high-precision dosing of the iodine. Because the coulometric Karl Fischer method is an **absolute determination**, no titer needs to be determined. It must only be ensured that the reaction generating the iodine runs with a 100% current efficiency. All of the reagents available today ensure this.

The endpoint indication is effected voltametrically by modulating an alternating current of constant strength to a double Pt electrode. This results in a voltage difference between the Pt wires. This is drastically reduced as soon as even the slightest amounts of free iodine are present. This circumstance is used for detecting the endpoint of the titration.

7.2 OMNIS Coulometer – Working with water standards

Certified water standards

Commercially available, certified water standards with water contents of 1.00 ± 0.003 mg/g and/or 0.10 ± 0.005 mg/g should be used for validating the instrument as a whole, integrated system.

i The 1.0 mg/g water standard is easier to handle and is therefore preferred.

Table 6 Recommended weighing ranges

1.0 mg/g water standard	0.2–2.0 g
0.1 mg/g water standard	0.5–5.0 g

7.3 OMNIS Coulometer – Sample addition

This chapter contains a few notes concerning sample addition. An exhaustive discussion of this topic is not possible here. Further notes can be found in the literature by the reagent manufacturers and in the following **Metrohm Application Bulletins**:

Bulletin no.	Title
No. 137	Coulometric water content determination according to Karl Fischer
No. 142	Karl Fischer water content determination in non-explosive gases
No. 145	Determination of low water contents in plastics using the KF oven method
No. 209	Coulometric water content determinations according to the Karl Fischer method in insulating oils, hydrocarbons, and their products

Values for the sample sizes

The sample weight should be small in order to be able to titrate as many samples as possible in the same electrolyte solution and to keep the titration time short. However, ensure that the sample contains at least 50 µg of H₂O. The following table helps you determine the appropriate sample size.

Table 7 Recommended sample sizes

Water content of the sample	Sample size	Resulting water content
10,000 ppm = 1%	10–100 mg	100–1,000 µg

Water content of the sample	Sample size	Resulting water content
1,000 ppm = 0.1%	100 mg–1 g	100–1,000 µg
100 ppm = 0.01%	1 g	100 µg
10 ppm = 0.001%	5 g	50 µg

Working with liquid samples

Liquid samples are added with a syringe. The samples can be injected two different ways:

- Use a syringe with a long needle that you immerse in the reagent during the injection.
- Use a syringe with a short needle and aspirate the last drop back into the needle.

The best way for you to determine the injected sample amount is to weigh the sample by difference.

Glass syringes should be used for the **determination of traces and validations**. Metrohm recommends obtaining these from a specialized syringe manufacturer.

Highly volatile samples and samples of low viscosity should be cooled before sampling. Doing so avoids losses while working. The syringe must, however, not be cooled directly, as condensation could be formed. For the same reason, no air may be aspirated into a syringe into which a cooled sample has been aspirated beforehand.

Samples of high viscosity can be thinned by heating. The syringe must be heated as well. The same target can be reached by diluting with suitable solvents. In this case, the water content of the solvent has to be determined and subtracted as a blank value.

If samples contain only **traces of water**, then the syringe has to be predried well. If possible, the syringe should be rinsed with the sample solution by filling in and discarding the solution several times.

Working with solid samples

Solid samples such as powders, pastes, fats, and oils are extracted or dissolved in a suitable solvent if possible. The resulting solution is injected, during which a blank value correction for the solvent must be carried out.

If no suitable solvent can be found for a solid sample, or if the sample reacts with the Karl Fischer reagent, then a Karl Fischer oven should be used.

8.2 Magnetic stirrer – Operation

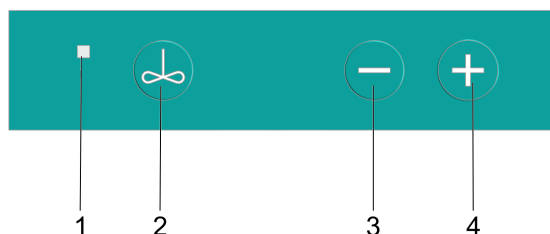


Figure 7 Magnetic stirrer – Control bar

1 Status display Multi-colored	2 On/Off (see "Switching the magnetic stirrer on and off", chapter 8.2.1, page 39)
3 Reduce stirring rate (see "Setting the magnetic stirrer", chapter 8.2.2, page 40)	4 Increase stirring rate (see "Setting the magnetic stirrer", chapter 8.2.2, page 40)

Other functions in the software

The following functions can be executed only with the OMNIS Software (see [OMNIS Help](#)):

- **Deactivate keys**
The magnetic stirrer can be operated only via the software.
- **Switch over the keys for the rod stirrer**
The keys on the magnetic stirrer operate the rod stirrer.
- **Set the stirring direction**

8.2.1 Switching the magnetic stirrer on and off

1 Switching the magnetic stirrer on

Press the  key.

The magnetic stirrer stirs with the most recently used stirring rate.

2 Switching the magnetic stirrer off

Press the  key once again.

The magnetic stirrer stops.



i If the magnetic stirrer is running at a high stirring rate, reduce the stirring rate before switching it off.

As an alternative, switch the magnetic stirrer on and off in the OMNIS Software under *Manual control*.

8.2.2 Setting the magnetic stirrer

The stirring rate can be adjusted in 15 steps.

Prerequisite:

The magnetic stirrer is switched on.

1 Increasing the stirring rate in steps

Press the  key.

Each pressing of the key increases the stirring rate by 1 step. The current stirring rate appears in the OMNIS Software under **Manual control**.

2 Reducing the stirring rate

Press the  key.

Each pressing of the key reduces the stirring rate by 1 step. The current stirring rate appears in the OMNIS Software under **Manual control**.

Alternatively, the stirring rate can also be set in the OMNIS Software under *Manual control*.

i The stirring direction can be exclusively set in the OMNIS Software under **Manual control**.

8.3 Reagent replacement

The electrolyte solutions must be replaced in the following cases:

- The titration cell is too full.
- The KF reagent has reached its capacity limit.
- The drift is too high and cannot be reduced by shaking the titration cell.
- A two-phase-mixture is formed in the titration cell. In this case it is also possible to aspirate only the sample phase.


Exhausted electrolyte solution is best disposed of by aspiration. An advantage is that the titration cell does not have to be disassembled. In addition, no humidity gets into the titration cell as it is not opened.

In the event of severe contamination, the titration cell can be rinsed with a suitable solvent which is also aspirated.

In the case of a generator electrode with diaphragm, the catholyte should be replaced once per week. Longer use can cause blackening and yellow precipitates in the cathode chamber. An unpleasant smell is also a sign of having used the catholyte for too long.

8.3.1 Reagent replacement with dosing drive and piston buret

When replacing reagent with a dosing drive and a piston buret, the used reagent is aspirated through the FEP tubing from the Karl Fischer titration cell into the piston buret. The used reagent is ejected from the piston buret into the waste bottle through the FEP tubing connected to the waste port.

 If necessary, clean the titration cell with multiple rinsing cycles.

The fresh reagent is dosed from the chemical bottle through the FEP tubing into the piston buret. The fresh reagent is dosed from the piston buret through the FEP tubing into the titration cell.

The aspiration tip is filled with fresh reagent after dosing. Dry air is drawn into the piston buret through the adsorber tube so that no liquid flows from the aspiration tip into the titration cell during the measurement. The dry air is ejected into the titration cell together with the residual liquids from the aspiration tip. This allows the titration cell to be completely dried before the measurement.

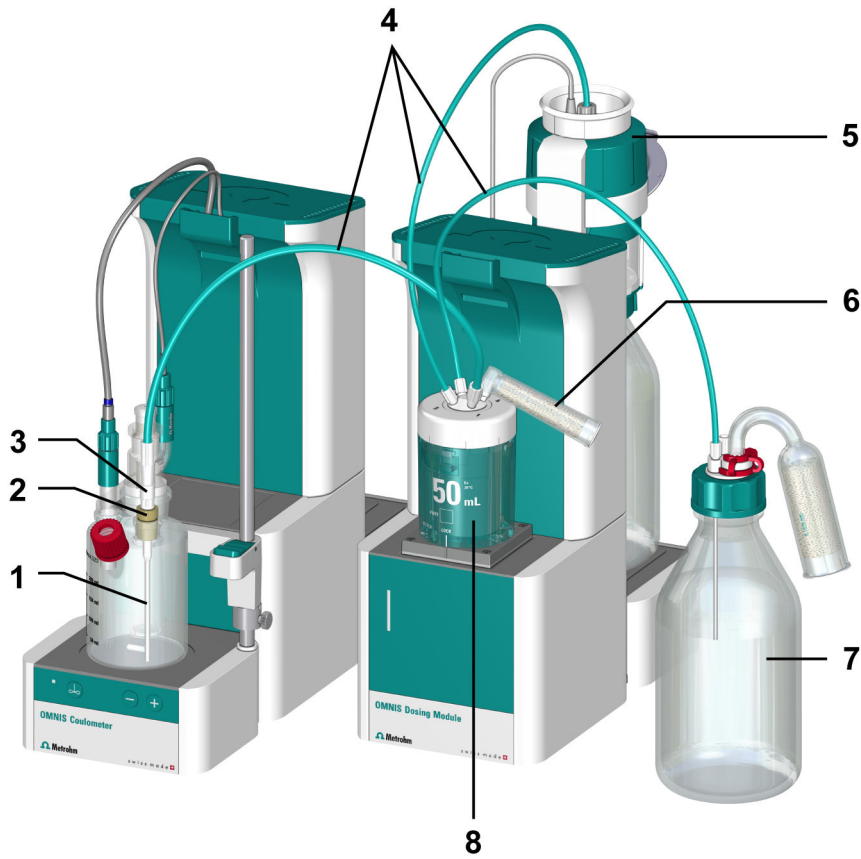


Figure 8 Reagent replacement with OMNIS Dosing Module – Example

<p>1 Antidiffusion tip (6.1543.200) without antidiffusion valve</p>	<p>2 Stoppers for reagent replacement (6.1446.060) with ground-joint sleeve (6.2713.000)</p>
<p>3 Adapter for reagent replacement (6.2730.030) with nipple and O-ring</p>	<p>4 FEP tubings (6.1805.100)</p>
<p>5 OMNIS Liquid Adapter (6.01600.010) on a bottle with KF reagent</p>	<p>6 Adsorber tube for a cylinder unit OMNIS (6.1619.020)</p>
<p>7 Waste bottle (6.1608.030)</p>	<p>8 Cylinder unit OMNIS 50 mL (6.01503.250)</p>

For a reagent replacement with an OMNIS Dosing Module, a tubing setup such as that shown in the figure is required. Proceed as follows:

Preparing the reagent replacement

Prerequisite:

- The ground-joint stopper has been removed from the ground-joint opening on the right.



Required accessories:

- *(see figure 8, page 42)*

1 Assembling the stopper

- Screw the nipple of the adapter with the O-ring onto the stopper.
- Remove the antidiffusion valve from the antidiffusion tip to obtain an aspiration tip.
- Slide the aspiration tip through the stopper.
- Place the ground-joint sleeve over the stopper.
- Insert the stopper together with the aspiration tip and the ground-joint sleeve into the right-hand ground-joint opening of the titration cell.
- Insert the aspiration tip into the titration cell until it touches the vessel base.

2 Connecting the titration cell with the cylinder unit OMNIS

- Screw the first FEP tubing onto the aspiration tip.
- Screw the other end of the FEP tubing onto the dosing port of the cylinder unit OMNIS.

3 Connecting the cylinder unit OMNIS with the waste bottle

- Screw the second FEP tubing onto the waste port of the cylinder unit OMNIS.
- Screw the other end of the FEP tubing onto the waste bottle to aspirate the used reagent from the titration cell and dose it through the cylinder unit OMNIS into the waste bottle.

4 Connecting the cylinder unit OMNIS with the Liquid Adapter

- Screw the third FEP tubing onto the fill port of the cylinder unit OMNIS.
- Screw the other end of the FEP tubing onto the Liquid Adapter to dose the fresh reagent through the cylinder unit OMNIS into the titration cell.

5 Mounting the adsorber tube

- Screw the adsorber tube onto the free port.



8.3.2 Reagent replacement with OMNIS Solvent Module

When replacing reagents with the OMNIS Solvent Module, the used reagent is aspirated through the PTFE tubing from the Karl Fischer titration cell and pumped into the waste bottle.

i If necessary, clean the titration cell with multiple rinsing cycles.

The fresh reagent is pumped into the titration cell via the OMNIS Solvent Module.

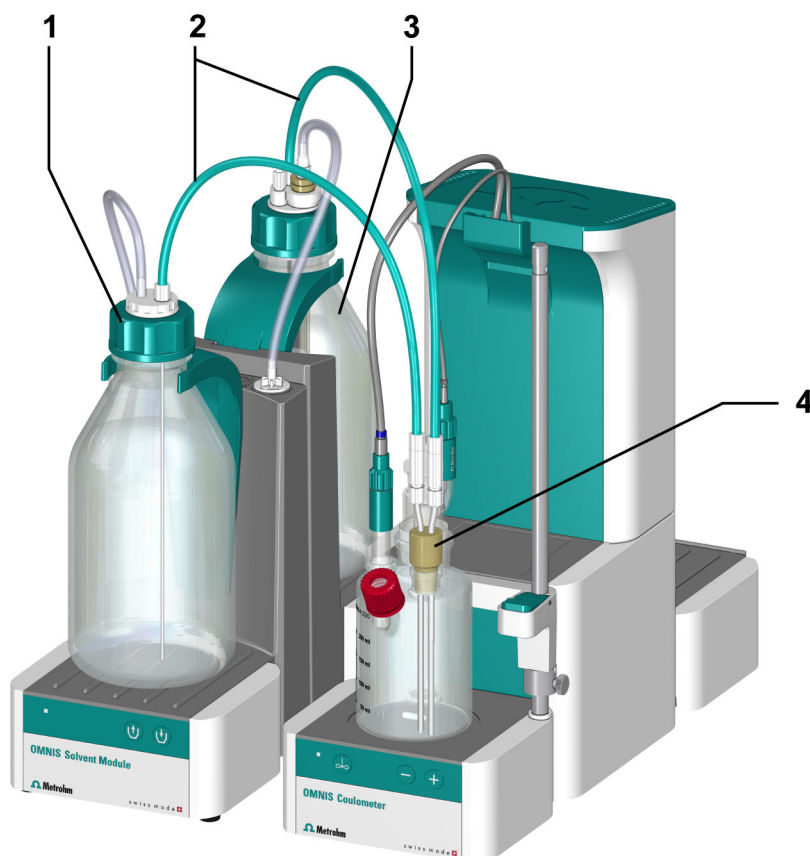


Figure 9 Reagent replacement with OMNIS Solvent Module

<p>1 Siphon Breaker (6.01600.200) on bottle with KF reagent</p>	<p>2 PTFE tubing (6.1805.200)</p>
<p>3 Waste bottle. (6.1608.030)</p>	<p>4 Adapter for reagent replacement (6.1446.220) with ground-joint sleeve (6.2713.000)</p>

For a reagent replacement with the OMNIS Solvent Module, a tubing setup such as that shown in the figure is required. Proceed as follows:

Preparing reagent replacement

Prerequisite:

- The ground-joint stopper has been removed from the ground-joint opening on the right.

Required accessories:

- *(see figure 9, page 44)*

1 Inserting the adapter

- Place the ground-joint sleeve over the adapter.
- Insert the adapter together with the ground-joint sleeve into the right-hand ground-joint opening of the titration cell with the aspiration tips facing downwards.

2 Connecting the titration cell with the waste bottle

- Screw the first PTFE tubing onto the closed aspiration tip.
- Screw the other end of the PTFE tubing onto the waste bottle to pump the used reagent into the waste bottle.

3 Connecting the titration cell with the Siphon Breaker

- Screw the second PTFE tubing onto the open aspiration tip.
- Screw the other end of the PTFE tubing onto the Siphon Breaker on the fresh reagent to pump the fresh reagent into the titration cell.

8.3.3 Manual reagent replacement

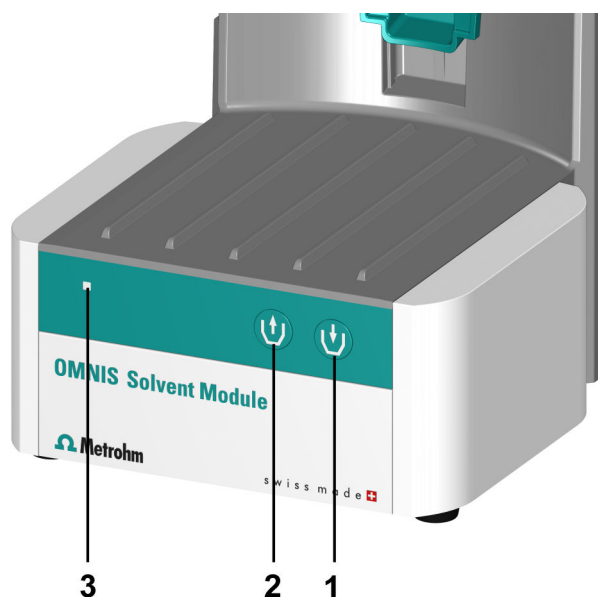



Figure 10 OMNIS Solvent Module – Indicators and controls

- | | |
|------------------------------------------------------------------------------|-------------------------------------------------------------------------|
| <p>1 Add key
Deliver liquid (Solvent) into the titration cell</p> | <p>2 Aspirate key
Aspirate waste from the titration cell</p> |
| <p>3 Status display
Multi-colored</p> | |

Prerequisite:

- OMNIS Solvent Module is connected.
- The solvent bottle, waste bottle and Karl Fischer titration cell are fully mounted and connected with the corresponding tubings.

1 Manually emptying the KF titration cell

Press the  key on the OMNIS Solvent Module:

The OMNIS Solvent Module starts aspirating waste out of the Karl Fischer titration cell into the waste bottle.

Different versions are possible:

- Long pressing (> 1 s): Aspirating takes place until the key is released. The pumping duration is saved.
- Short pressing (\leq 1 s): The aspiration takes place during the saved pumping duration. Press the key again to stop the procedure prematurely.


2 Manually filling the KF titration cell

Press the  key on the OMNIS Solvent Module:

The OMNIS Solvent Module starts aspirating waste out of the Karl Fischer titration cell into the waste bottle.

Different versions are possible:

- Long pressing (> 1 s): Aspirating takes place until the key is released. The pumping duration is saved.
- Short pressing (≤ 1 s): The aspiration takes place during the saved pumping duration. Press the key again to stop the procedure prematurely.

 The reagent replacement can also be carried out automatically via the OMNIS Software. Additional information under <https://www.metrohm.com>.

**WARNING****Health hazards from electrical potential.**

Severe injuries with possibly fatal consequences.

- Operate the product only if it is in perfect condition. The housing must also be intact.
- Only use the product with the covers fitted.
- Protect live components (e.g. power supply unit, power cord, connection sockets) against moisture.
- Always have maintenance work and repairs on electrical components carried out by a regional Metrohm service representative.

Prerequisite:

- The product is switched off and disconnected from the energy supply.

Required accessories:

- Cleaning cloth (soft, lint-free)
- Water or ethanol

1 Clean the surface with a damp cloth. Remove persistent contamination with ethanol.

2 Wipe the surface with a dry cloth.

3 Clean the connectors with a dry cloth.

10 Troubleshooting

Messages on malfunctions and errors are displayed in the control software or in the embedded software (e.g. on the display of an instrument) and contain the following information:

- Descriptions of causes of malfunctions (e.g. jammed drive)
- Descriptions of problems with the control (e.g. missing or invalid parameter)
- Information on how to solve the problem

System components with status display elements also indicate malfunctions and errors with a red flashing LED.

Troubleshooting on the product is often only possible with the control software or the embedded software (e.g. initializing, moving to a defined position).

See also

System – Signals (chapter 3.4, page 15)

10.1 Karl Fischer titration

Problem	Cause	Remedy
The drift is very high during conditioning.	The titration cell is leaking.	<ul style="list-style-type: none"> ▪ Check the seals and the septum. Replace if necessary. ▪ Replace the molecular sieve.
The drift becomes greater after each titration.	The sample releases water very slowly.	<ul style="list-style-type: none"> ▪ Adjust the method. ▪ Add solubility promoter. ▪ Increase the temperature (possibly using a KF oven). ▪ See technical literature.
	A side reaction is taking place.	<ul style="list-style-type: none"> ▪ Use special reagents. ▪ Adjust the method (increase/decrease the temperature, external extraction). ▪ See technical literature.
	The pH value is no longer in the optimum range.	Add buffer (see technical literature).


Problem	Cause	Remedy
The titration does not finish.	The titration cell is leaking.	<ul style="list-style-type: none"> ▪ Check the seals and the septum. Replace if necessary. ▪ Replace the molecular sieve.
	The stop criterion is unsuitable.	Adjust the control parameters (see manual/help of the software used): <ul style="list-style-type: none"> ▪ Increase the stop drift. ▪ Select a short delay time.
	See also: The drift becomes greater after each titration.	
The sample is overtitrated.	The amount of methanol in the working medium is too low.	<ul style="list-style-type: none"> ▪ Replace the working medium. ▪ Reduce the amount of solubility promoter, if working with solvent mixtures, see technical literature.
	The electrode may be covered.	Wipe off the electrode with ethanol or a suitable solvent.
The solution becomes darker after each titration.		Replace the working medium.
	The electrode may be covered.	Wipe off the electrode with ethanol or a suitable solvent.
	The electrode has a short circuit.	<ul style="list-style-type: none"> ▪ Check the Pt wires. ▪ Activate the electrode check.
The endpoint is reached too quickly.	The dosing rate outside the control range is too high.	Select the user-defined titration rate and reduce the dosing rate (see manual/help of the software used).



10.2 Forcing a shutdown

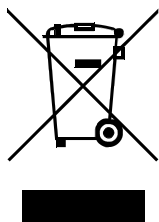
Prerequisite:

The OMNIS main instrument cannot be switched off.

- 1 Press the on/off switch  for 8 seconds until the acoustic signal is heard in short intervals.

The acoustic signal sounds for 2 seconds. The status display goes out and the OMNIS main instrument is switched off.

11 Disposal



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



12 Technical specifications

12.1 Ambient conditions

Nominal function range	+5 to +45 °C	at max. 80% relative humidity, non-condensing
Storage	+5 to +45 °C	at max. 80% relative humidity, non-condensing
Altitude / Pressure range	Max. 3,000 m.a.s.l. / min. 700 mbar	
Overvoltage category	II	
Pollution degree	2	

12.2 OMNIS Coulometer – Energy supply

Nominal voltage range	100–240 VAC ± 10%	
Nominal frequency range	50–60 Hz ± 3%	
Power consumption	max. 100 W	
Protection		
<i>Internal fuse</i>	4 ATH	cannot be replaced by the user



12.3 OMNIS Coulometer – Dimensions

Measurements

<i>Width</i>	142 mm
<i>Height</i>	358 mm
<i>Depth</i>	
Without magnetic stirrer	284 mm
With magnetic stirrer	400 mm

Weight

<i>Type</i>	
Without magnetic stirrer	4.4 kg
With magnetic stirrer	5.1 kg

12.4 Magnetic stirrer – Dimensions

Measurements

<i>Width</i>	142 mm
<i>Height</i>	70 mm
<i>Depth</i>	116 mm

Weight	700 g
---------------	-------

12.5 OMNIS Coulometer – Housing

Materials

<i>Cover</i>	PET	poly(ethylene terephthalate)
<i>Back panel</i>	AW-5754 H12/H22	aluminum, coated
<i>Base</i>	1.4301	stainless steel
<i>Enclosure</i>	PBT	poly(butylene terephthalate)

Magnetic stirrer – Housing



Front foils

PET

poly(ethylene terephthalate), mat

IP degree of protection

IP 40

12.6 Magnetic stirrer – Housing

Materials

Cover

PBT

Poly(butylene terephthalate)

Base

Chromium steel sheet

Enclosure

PBT

Poly(butylene terephthalate)

Front foils

PET

Poly(ethylene terephthalate)

IP degree of protection

IP 40

12.7 Operation specifications

Control bar

Stirrer, rotational speed

12.8 Operation specifications

Key

On/Off

Control bar

Stirrer, rotational speed

12.9 Connectors specifications

Energy supply

Socket

via power connection

IEC 60320, type C14,
10 A

Power cord

Length max. 2 m

Number of conductors

3

with protective ground

Conductor cross-section

min. 0.75 mm² / 18 AWG

Plug

Instrument side

IEC 60320, type C13,
10 A

Building side

country-specific

MDL

Metrohm Device Link

4 connectors

HID

Human Interactive Device

1 connector

LAN

Local Area Network

Type

Ethernet CAT 6

Socket

RJ45

shielded

Cable type

min. F/FTP

shielded

Cable length

max. 10 m

from Metrohm accessories ([see "Displaying the accessories", chapter 1.5, page 3](#))

Internal measuring interface

INPUT 1

Socket

round plug 7-pin,
size 0, 45°

Potentiometric

pH, ISE, Redox

Measuring input for
potentiometric electrodes

Display specifications



Temperature	Temp.	Measuring input for temperature sensors of the Pt1000 or NTC type for automatic temperature compensation
Polarizer	Pol.	Measuring input for polarizable electrodes
<i>INPUT 2</i>		
Socket		round plug 7-pin, size 0, 45°
Potentiometric	pH, ISE, Redox	Measuring input for potentiometric electrodes
Temperature	Temp.	Measuring input for temperature sensors of the Pt1000 or NTC type for automatic temperature compensation
<i>GENERATOR</i>		
	Socket	Round plug 2-pin
	Current generator output	For reagent generation

12.10 Display specifications

Status display	LED	multi-colored
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12.11 Current generator specifications

Low voltage generator (for bromine 1492)

<i>Current range</i>	0.5– 60.0 mA
<i>Voltage range</i>	0.0– 29.0 V

High voltage generator (for KFC water and BRC bromine index)

<i>Current range</i>	50.0– 400.0 mA
<i>Low voltage range</i>	0.0– 29.0 V
<i>High voltage range</i>	0.0– 39.0 V

Iodine production for Karl Fischer water determination

<i>Determination range</i>	0.01– 200.0 mg H ₂ O	Recommended water quantity
<i>Resolution</i>	0.1 µg H ₂ O	
<i>Titration rate</i>	max. 2.24 mg H ₂ O/min	
<i>Reproducibility</i>	±3 µg H ₂ O	at 10 µg– 1000 µg H ₂ O
	≤0.3%	Sample: Standard from the reagent manufacturer >1000 µg H ₂ O

12.12 Measurement specifications

Potentiometric

<i>Measuring range</i>	–2,400 to +2,400 mV –13 to +20 pH	
<i>Resolution</i>	1.56 µV 0.001 pH	
<i>Measuring accuracy</i>	±0.5 mV ±0.003 pH	in the measuring range –2,000 mV to +2,000 mV
<i>Input resistance</i>	≥ 1*10 ¹² Ω	
<i>Offset current</i>	≤ ±1*10 ⁻¹² A	

Temperature

<i>Pt1000</i>		
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
<i>NTC 30 kOhm</i>		
Measuring range	–5 to +250 °C	
Measuring resolution	approx. 0.002 °C	

Measurement specifications



Measuring accuracy	$\pm 0.6\text{ }^{\circ}\text{C}$	in the measuring range $+10.0\text{ }^{\circ}\text{C}$ to $+40.0\text{ }^{\circ}\text{C}$
--------------------	-----------------------------------	----------------------------------------------------------------------------------------------

Polarizer

I_{pol DC}

Polarization current	-200.0 to $+200.0\text{ }\mu\text{A}$	adjustable in $0.5\text{ }\mu\text{A}$ steps
Measuring range	$-2,400$ to $+2,400\text{ mV}$	
Measuring resolution	0.1 mV	

I_{pol AC}

Polarization current	$5\mu\text{A}$, $10\mu\text{A}$, $20\mu\text{A}$, $30\mu\text{A}$	Effective values
Measuring range	0 — $+1,700\text{ mV}$	Effective value
Measuring resolution	0.1 mV	Effective value
Frequency	10 Hz	

Upol DC

<i>Polarization voltage</i>	$-2,000\text{ mV}$ to $+2,000\text{ mV}$	adjustable in 5 mV steps
<i>Measuring range</i>	$-200.0\text{ }\mu\text{A}$ to $+200.0\text{ }\mu\text{A}$	
<i>Measuring resolution</i>	$0.01\text{ }\mu\text{A}$	

Load of the measuring input I_{pol}

<i>R_{L max.} ±10μA</i>	$240\text{ k}\Omega$
<i>R_{L max.} ±50μA</i>	$48\text{ k}\Omega$
<i>R_{L max.} ±100μA</i>	$24\text{ k}\Omega$

Load of the measuring input Upol

<i>R_{L min.} ±300 mV</i>	$1.5\text{ k}\Omega$
<i>R_{L min.} ±600 mV</i>	$3\text{ k}\Omega$
<i>R_{L min.} ±1,000 mV</i>	$5\text{ k}\Omega$

Measuring accuracy



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

Reference conditions

<i>Relative humidity</i>	≤ 60%
<i>Ambient temperature</i>	+25 °C (±3 °C)
<i>Instrument status</i>	

min. 30 minutes in operation

12.13 Magnetic stirrer – Specifications

Adjustment range for rotational speed	+1 to +15	Rotation in counter-clockwise direction (seen from above)
	-1 to -15	Rotation in clockwise direction (seen from above)
Rotational speed change per step	120 rpm	
Maximum rotational speed	1,800 rpm	
Lengths of stirring bar	8, 12, 16, 25, 30 mm	