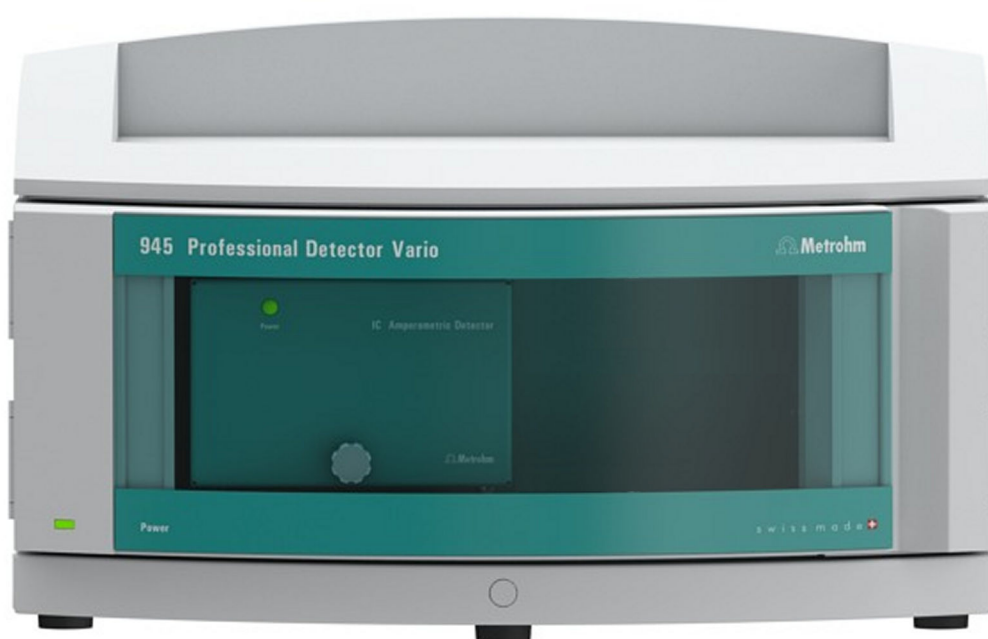


# 945 Professional Detector Vario



## 945 Professional Detector Vario – Amperometry

### Manual

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Metrohm AG  
CH-9100 Herisau  
Switzerland  
Phone +41 71 353 85 85  
Fax +41 71 353 89 01  
info@metrohm.com  
www.metrohm.com

# **945 Professional Detector Vario**

## **945 Professional Detector Vario – Amperometry**

2.945.0020

### **Manual**

Technical Communication  
Metrohm AG  
CH-9100 Herisau  
techcom@metrohm.com

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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.

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# 1 Introduction

## 1.1 Instrument description

The **945 Professional Detector Vario – Amperometry** is an intelligent stand-alone detector equipped with an amperometric detector.

As a stand-alone detector, it can be combined with instruments such as those of the 940 Professional IC Vario family for which all available detector connectors have already been assigned to conductivity detectors (AnCat systems or other multi-channel systems) and be used for the determination of electroactive substances in the mobile phase.

The 945 Professional Detector Vario – Amperometry enables applications that require amperometric detection also with instruments of the 930 Compact IC Flex family and the 883 Basic IC plus, which are equipped with only one detector connector, which is normally occupied by a conductivity detector.

The 945 Professional Detector Vario – Amperometry is a stand-alone detector that combines the advantages of the IC Amperometric Detector with the combination possibilities provided by 940 Professional IC Vario instruments. It is directly controlled by the MagIC Net software.

The 942 Extension Module Vario, 891 Professional Analog Out and 800 Dosinos, Remote Boxes, etc. can all be operated through the 945 Professional Detector Vario – Amperometry. This opens up the flexibility of Metrohm IC systems considerably.

The instrument is comprised of the following modules:

### **Amperometric detector**

With the 945 Professional Detector Vario – Amperometry, electroactive substances can be determined in the mobile phase of an IC system. Determination makes use of amperometric methods, which combine outstanding sensitivity with a high degree of selectivity. The installed potentiostat generates the voltages for the direct current amperometry (DC), for the pulse amperometry (PAD) and the flexible integrated pulse amperometry (flexIPAD) as well as for the recording of cyclovoltammograms. The installed preheating capillary ensures a constant eluent temperature on the cell.

## 1.2 Intended use

The 945 Professional Detector Vario – Amperometry is used for the amperometric detection in an IC system. Its function is to make precise determinations of electroactive substances in the mobile phase of an IC system or of a general liquid chromatography system.

The present instrument is used for working with chemicals and flammable samples. Therefore, the use of the 945 Professional Detector Vario – Amperometry requires the user to have basic knowledge and experience in handling toxic and caustic substances. Knowledge regarding the application of fire prevention measures prescribed for laboratories is also mandatory.

## 1.3 Safety instructions

### 1.3.1 General notes on safety



#### WARNING

---

Operate this instrument only according to the information contained in this documentation.

This instrument left the factory in a flawless state in terms of technical safety. To maintain this state and ensure non-hazardous operation of the instrument, the following instructions must be observed carefully.

### 1.3.2 Electrical safety

The electrical safety when working with the instrument is ensured as part of the international standard IEC 61010.



#### WARNING

---

Only personnel qualified by Metrohm are authorized to carry out service work on electronic components.



### WARNING

Never open the housing of the instrument. The instrument could be damaged by this. There is also a risk of serious injury if live components are touched.

There are no parts inside the housing which can be serviced or replaced by the user.

### Supply voltage



### WARNING

An incorrect supply voltage can damage the instrument.

Only operate this instrument with a supply voltage specified for it (see rear panel of the instrument).

### Protection against electrostatic charges



### WARNING

Electronic components are sensitive to electrostatic charges and can be destroyed by discharges.

Do not fail to pull the power cord out of the power socket before you set up or disconnect electrical plug connections at the rear of the instrument.

The device is to be operated only with the door closed.

## 1.3.3 Tubing and capillary connections



### CAUTION

Leaks in tubing and capillary connections are a safety risk. Tighten all connections well by hand. Avoid applying excessive force to tubing connections. Damaged tubing ends lead to leakage. Appropriate tools can be used to loosen connections.

Check the connections regularly for leakage. If the instrument is used mainly in unattended operation, then weekly inspections are mandatory.

### 1.3.4 Flammable solvents and chemicals

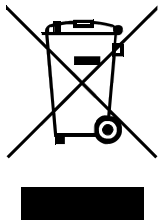


#### WARNING

All relevant safety measures are to be observed when working with flammable solvents and chemicals.

- Set up the instrument in a well-ventilated location (e.g. fume cupboard).
- Keep all sources of flame far from the workplace.
- Clean up spilled liquids and solids immediately.
- Follow the safety instructions of the chemical manufacturer.

### 1.3.5 Recycling and 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.

## 1.4 About the documentation



#### CAUTION

Read through this documentation carefully before putting the device into operation. The documentation contains information and warnings which the user must follow in order to ensure safe operation of the device.

### 1.4.1 Content and scope







This document describes the **945 Professional Detector Vario – Amperometry**, its assembly and connection to the IC instrument, as well as the installation, operation and maintenance of the individual components. Technical specifications, troubleshooting and information concerning scope of delivery and optional accessories make up the rest of the manual.

You will find additional information on the installation and maintenance of the IC instrument and the Sample Processor in the respective manuals.

Additional information on the configuration and operation with MagIC Net can be found in the "*MagIC Net Tutorial*" or in the MagIC Net online help.

## 1.4.2 Symbols and conventions

The following symbols and formatting may appear in this documentation:

(5-12)	<b>Cross-reference to figure legend</b> The first number refers to the figure number, the second to the instrument part in the figure.
<b>1</b>	<b>Instruction step</b> Carry out these steps in the sequence shown.
<b>Method</b>	<b>Dialog text, parameter</b> in the software
<b>File ► New</b>	Menu or menu item
<b>[Next]</b>	<b>Button or key</b>
	<b>WARNING</b> This symbol draws attention to a possible life-threatening hazard or risk of injury.
	<b>WARNING</b> This symbol draws attention to a possible hazard due to electrical current.
	<b>WARNING</b> This symbol draws attention to a possible hazard due to heat or hot instrument parts.
	<b>WARNING</b> This symbol draws attention to a possible biological hazard.
	<b>CAUTION</b> This symbol draws attention to possible damage to instruments or instrument parts.
	<b>NOTE</b> This symbol highlights additional information and tips.



## 2 Overview of the device

### 2.1 Front

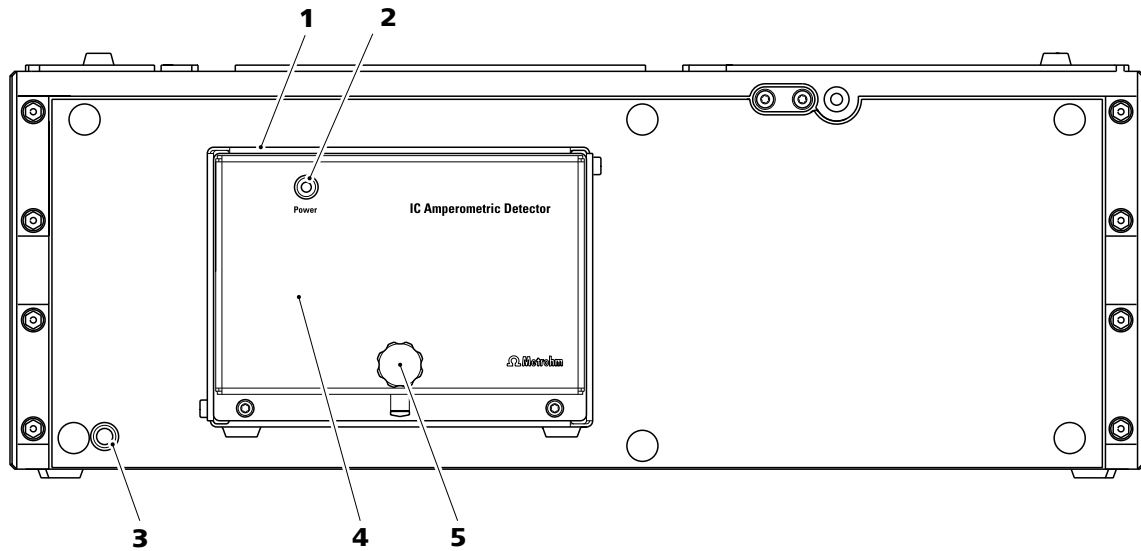


Figure 1 Front – Front cover attached

<p><b>1 Amperometric detector</b> Permanently installed.</p>	<p><b>2 Power LED</b> Amperometric detector standby indicator.</p>
<p><b>3 Power LED</b> Instrument standby indicator.</p>	<p><b>4 Front cover</b> For the amperometric detector.</p>
<p><b>5 Knurled screw</b> For removing the front cover.</p>	

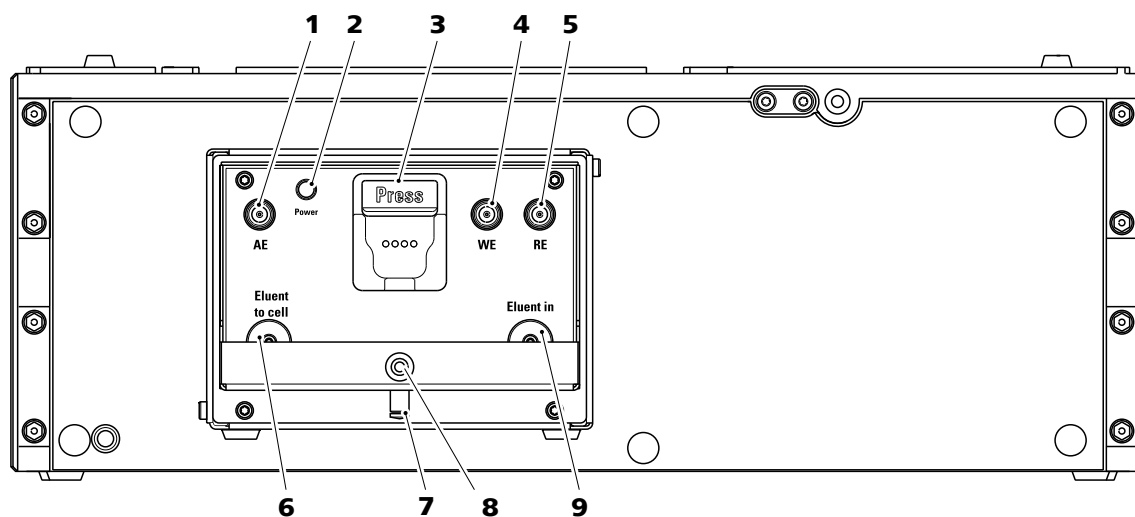


Figure 2 Front – Front cover removed

**1 AE connection socket**

For connecting the auxiliary electrode.

**3 Cell holder**

With chip for the automatic detection of the measuring cell.

**5 RE connection socket**

For connecting the reference electrode.

**7 Drain nozzle**

For draining liquid from the tray. Plugged with a stopper.

**9 Coupling**

For connecting a detector inlet capillary. Labeled *Eluent in*.

**2 Power LED**

Amperometric detector standby indicator.

**4 WE connection socket**

For connecting the working electrode.

**6 Coupling**

For connecting a connection capillary to the measuring cell. Labeled *Eluent to cell*.

**8 Thread**

For the knurled screw used for fastening the front cover.



## 2.2 Rear

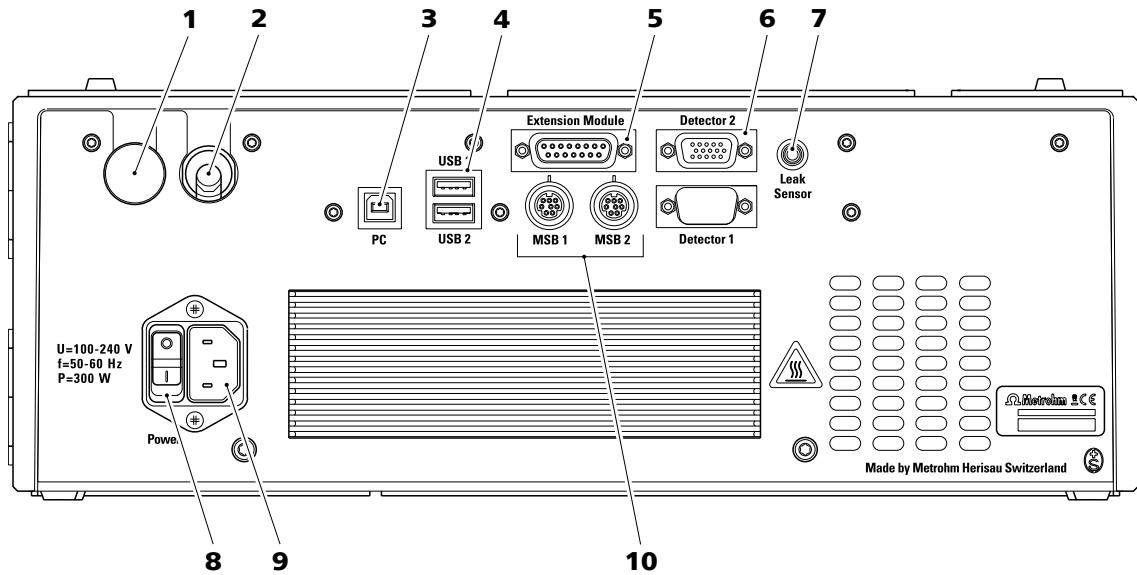


Figure 3 Rear

### 1 Stopper

Closes off the unused cable feed-through.

### 3 PC connection socket

For connecting the instrument to the computer with the USB cable (6.2151.020).

### 5 Extension Module connection socket

For connecting a 942 Extension Module Vario or an 891 Professional Analog Out. Labeled *Extension Module*.

### 7 Leak sensor connection socket

For connecting the leak sensor connection cable, labeled *Leak Sensor*.

### 9 Power socket

For connecting the power supply cable (6.2122.0x0).

### 2 Cable feed-through

Output for the detector cable.

### 4 USB connection sockets

Two USB connection sockets, labeled *USB 1* and *USB 2*.

### 6 Detector connection sockets

For the connection of the installed detector, labeled *Detector 1* and *Detector 2*. The detector connection sockets which are not used must be covered with a lid.

### 8 Power switch

For switching the instrument on and off.

### 10 MSB connection sockets

Two MSB connection sockets (labeled *MSB 1* and *MSB 2*) for connecting MSB devices. (MSB = Metrohm Serial Bus)

## 3 Installation

### 3.1 Base tray and bottle holder

#### 3.1.1 Basic information on base tray and bottle holder

The base tray (6.2061.110) and bottle holder (6.2061.100) protect IC instruments from dust, dirt and leaking fluids. The supply bottles for eluent and auxiliary solutions can be positioned neatly on the bottle holder.

In a complex IC system, several different instruments may be used, such as an analyzer, an extension module and a detector. These instruments can be set up in one or more stacks. We recommend that a base tray and bottle holder be mounted for each stack of IC instruments.

The bottle holder and base tray must be removed or set up if one of the following instruments is to be mounted on or under a 940 Professional IC Vario:

- One or more 942 Extension Module Vario
- One 944 Professional UV/VIS Detector Vario
- One 945 Professional Detector Vario
- Or another instrument with the same-sized footprint

#### 3.1.2 Mounting base tray and bottle holder (optional)

The base tray and bottle holder come fully assembled on a new ion chromatograph. To install an Extension Module on the ion chromatograph, remove the bottle holder and put it back on top of the topmost instrument. To install an Extension Module under the ion chromatograph, remove the base tray and set it under the lowest instrument.

##### 3.1.2.1 Removing/mounting the base tray

Remove the base tray to install another instrument under the IC instrument.



#### CAUTION

##### Do not pinch capillaries or leak sensor cables

Capillaries are fed through the guide ducts between the base tray and the instrument. Pinches in the leak sensor cable or the capillaries may lead to malfunctions.

- Unplug the leak sensor cable before you remove the base tray.
- Remove all the capillaries from the capillary ducts before you remove the base tray.

### Removing the base tray

#### Prerequisites

- The instrument is switched off.
- The bottle holder is cleared.
- All of the cable connections on the rear have been disconnected.
- The capillaries are removed from the guide ducts between the instrument and the base tray.
- There are no loose parts in the instrument.

#### Accessories

- 3 mm hex key (6.2621.100)

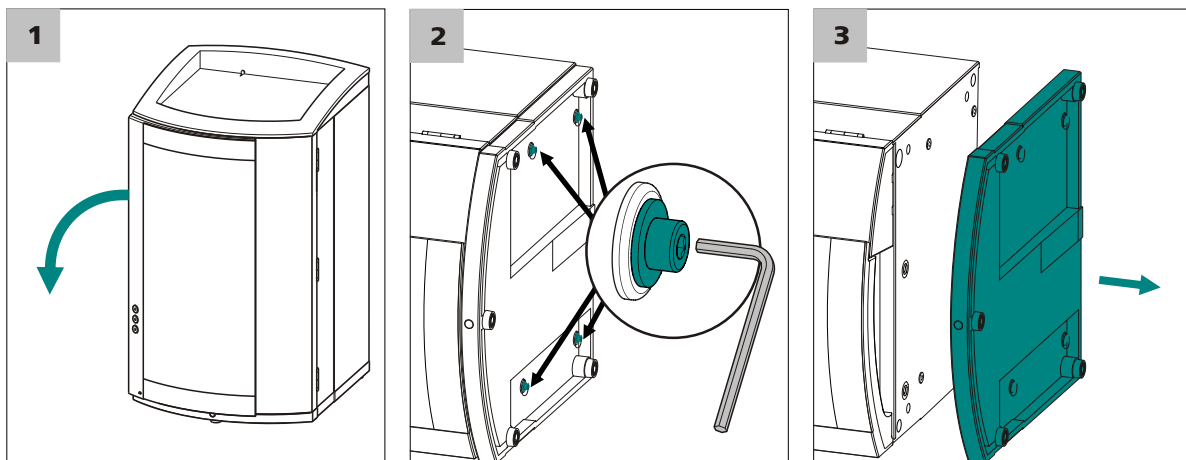


Figure 4 Removing the base tray

- 1** Tilt the instrument sideways and lay it down flat.
- 2** Loosen the 4 cylinder screws with the 3 mm hex key. Remove the cylinder screws and their washers.
- 3** Remove the base tray.

Always mount the base tray under the lowermost instrument of the stack.

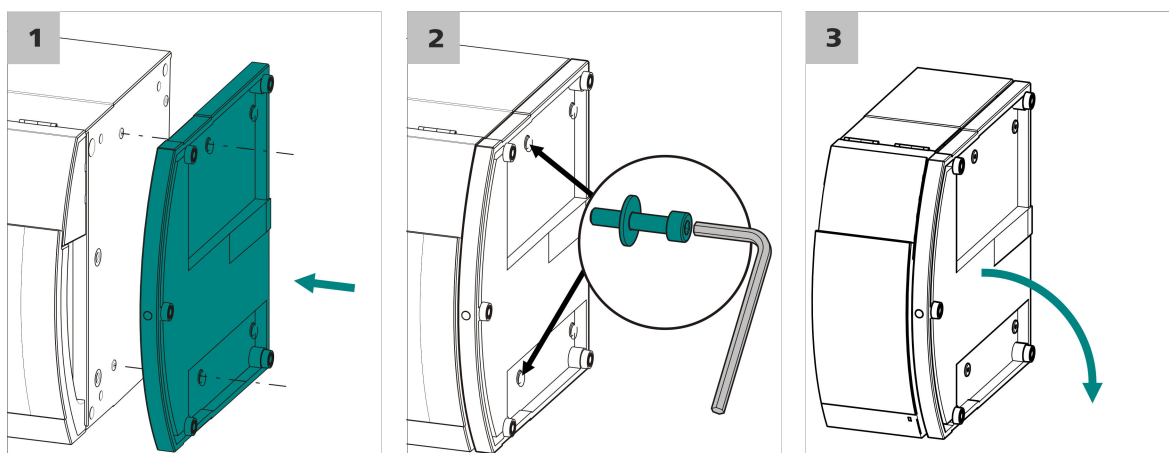
### Mounting the base tray

#### Prerequisites

- The instrument is switched off.
- The bottle holder is cleared.
- All of the cable connections on the rear have been disconnected.
- There are no loose parts in the instrument.
- The instrument is lying on its side, and the bottom surface is visible.

### Accessories

- 3 mm hex key (6.2621.100)



- 1** Mount the base tray in such a way that the openings in the base tray match exactly the screw threads in the bottom of the instrument.
- 2** Slide the washers onto the cylinder screws. Insert the cylinder screws with the washers and tighten them with the 3 mm hex key.
- 3** Set the instrument back up on the base tray.

Stack other instruments in the required order. Mount the bottle holder (6.2061.100) onto the topmost instrument on the stack (*see "Mounting the bottle holder", page 12*).

#### 3.1.2.2 Removing/mounting the bottle holder

Remove the bottle holder if you want to install another instrument onto the IC instrument.

##### Removing the bottle holder

##### Prerequisites

- The instrument is switched off.
- The bottle holder is cleared.
- Drainage tubing is disconnected from the drainage tubing connection of the bottle holder.
- The capillaries are removed from the guide ducts between the instrument and the bottle holder.

##### Accessories

- 3 mm hex key (6.2621.100)

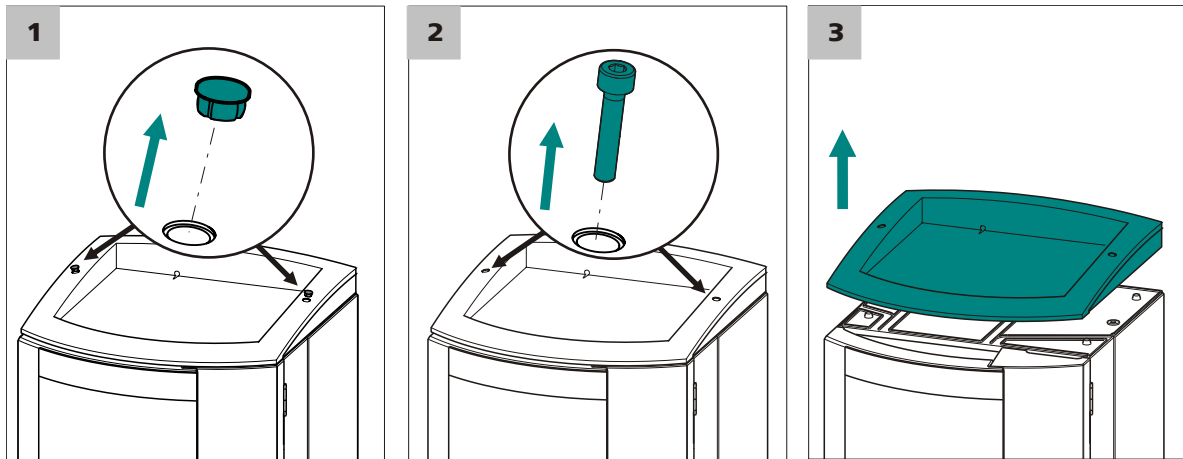


Figure 5 Removing the bottle holder

- 1 Remove the 2 covering stoppers.
- 2 Loosen the 2 cylinder screws with the 3 mm hex key and remove them.
- 3 Remove the bottle holder.

Stack other instruments in the required order. Mount the bottle holder (6.2061.100) onto the topmost instrument on the stack.

### Mounting the bottle holder

#### Prerequisite

- The instrument is switched off.

#### Accessories

- 3 mm hex key (6.2621.100)

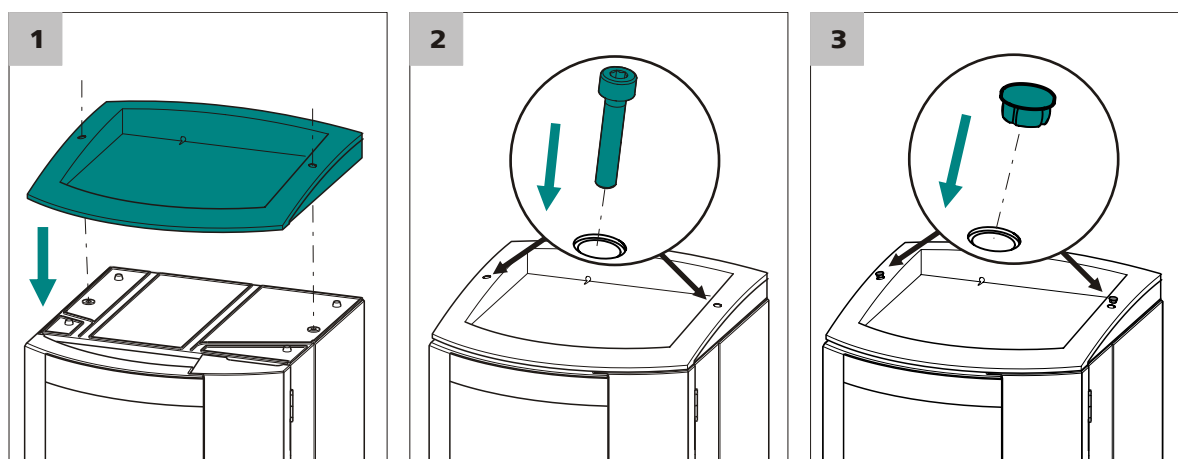


Figure 6 Mounting the bottle holder

- 1 Mount the bottle holder onto the topmost instrument in such a way that the openings in the bottle holder exactly match the screw threads on the top surface of the instrument.
- 2 Insert the 2 cylinder screws and tighten them with the 3 mm hex key.
- 3 Insert both covering stoppers.

After attaching the bottle holder, restore all connections that were loosened at the beginning of the process.

### Restoring the loosened connections

- 1 Plug in all necessary USB cables.
- 2 Plug in all necessary MSB cables.
- 3 Plug in the power cord.
- 4 Mount the drainage tubing again (*see manual of the IC instrument*).  
A longer section of silicone tubing (6.1816.020) may have to be cut to size and mounted (*see also the manual for the IC instrument*).
- 5 If one of the instruments in the stack is equipped with a leak sensor connection socket, connect the leak sensor (*see manual of the IC instrument*).
- 6 Restore any capillary connections that may have been removed.

## 3.2 Connecting the instrument to a computer



### NOTE

If the instrument is connected to the computer, then it must be switched off.

#### Accessories

For this step, you need the following accessories:

- USB connecting cable (6.2151.020)

### Connecting the USB cable

- 1 Insert the USB cable into the connection socket on the rear of the instrument labeled *PC*.
- 2 Insert the other end into a USB port on the computer.

## 3.3 Connecting the instrument to the power grid



### 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.

### Connecting the power cord

#### Accessories

Power cord with the following specifications:

- Length: max. 2 m
- Number of cores: 3, with protective conductor

- Instrument plug: IEC 60320 type C13
- Conductor cross-section 3x min. 1.0 mm<sup>2</sup> / 18 AWG
- Power plug:
  - according to customer requirement (6.2122.XX0)
  - min. 10 A

**NOTE**

Do not use a not permitted power cord!

**1 Plugging in the power cord**

- Plug the power cord into the instrument's power socket.
- Connect the power cord to the power grid.

## 3.4 Amperometric detector

The following tasks are part of the installation of the amperometric detector:

- Inserting the working electrode and the reference electrode into the measuring cell (see the measuring cell manual).
- Connecting the capillaries to the preheating capillary or directly to the measuring cell.
- Inserting the measuring cell into the detector.
- Deaerating the measuring cell.
- Connecting the electrode cables.
- Attaching the front cover.

Because not only the capillaries but also the electrode cables must be tested prior to their first use, none of these installation tasks are carried out until the time of the first start-up.



## 2 Connecting the electrode connection cables to the detector

(unless they are already connected)

- Plug the straight plug of the working electrode connection cable (red sleeve) into the **WE** socket of the detector.
- Plug the straight plug of the reference electrode connection cable (black sleeve) into the **RE** socket of the detector.
- Plug the straight plug of the auxiliary electrode connection cable (blue sleeve) into the **AE** socket of the detector.

## 3 Inserting the dummy cell

- Place the dummy cell into the tray of the detector.
- Attach the front cover.



### NOTE

The metal parts of the cable plugs must not touch the front cover.

## 4 Adjusting settings in MagIC Net

In the **Method** program part, create a new method for the instrument test with the dummy cell.

- Select the detector and add it as a new device.
- Select the **DC** mode.
- Set the following parameters for the DC mode:
  - **DC potential: 0.8 V**
  - **Range: Auto**
  - **Damping: off**
- Add an analysis for the detector channel **Current**.
- Add the entry **Current ► Start data acquisition** in the Time program subwindow.
- Save the method.

In the **Workplace** program part:

- Load the method.
- In the **Watch window**, display the **Current** channel with at least three decimal places.

## 5 Carrying out the test

In the **Manual** program part:



- On the tab of the detector, switch on the dummy cell with **[Apply]**.  
After no more than one minute, the detector signal should level off at  $2.667 \text{ nA} \pm 7\%$ . Noise should not exceed  $0.005 \text{ nA}$ .
- Switch the dummy cell off with **[Cell Off]**.  
With the dummy cell switched off and the detector hardware still running, the signal should drop below an absolute value of  $1 \text{ nA}$ , and noise should be limited to the third decimal place.  
Exactly even signals may indicate that new detector data is not correctly transmitted.

#### 6 Removing the dummy cell

- Pull out the electrode connection cables from the connectors **AE**, **WE** and **RE** of the dummy cell.
- Remove the dummy cell from the tray.

The dummy cell incorporates a resistor ( $300 \text{ M}\Omega$ ) and a capacitor ( $100 \text{ nF}$ ) connected in parallel. If, in DC mode, a potential of  $0.8 \text{ V}$  is applied, then a current of  $2.667 \text{ nA} (\pm 7\%)$  is measured in the dummy cell. The capacitor simulates a well-working measuring cell.

## 4.2 Testing the leak sensor

The leak sensor should not respond during the start-up. If the leak sensor nevertheless does respond during the start-up, you will find information for eliminating the problem in Chapter (*see chapter 6, page 30*).

To check whether the leak sensor is functioning, proceed as follows:

### Testing the leak sensor

- 1 Hold a cloth moistened with eluent or tap water on the two contacts of the leak sensor .

The leak sensor of the detector responds.

If the leak sensor does not respond, please request Metrohm Service.

## 4.3 Testing the preheating capillary

The amperometric detector is equipped with a preheating capillary in its interior to ensure that the eluent flows through the measuring cell at a constant temperature. The preheating capillary need not, however, always be connected. If the ambient conditions are optimal, then the measuring results can be sufficiently accurate, even without the use of the preheating capillary.



### CAUTION

The preheating capillary may not be used when working with highly flammable liquids.

The preheating capillary must be free of both leaks and blockages.

To check whether the preheating capillary is free of both leaks and blockages, proceed as follows:

### Testing the preheating capillary

#### 1 Connecting the detector inlet capillary

Use a pressure screw (6.2744.014) to fasten the detector inlet capillary to the **Eluent in** connector on the detector.

#### 2 Adjusting the settings in MagIC Net

- In the program part **Manual** of MagIC Net, set the maximum pressure of the high-pressure pump to 5 MPa.
- Set the flow rate to 0.1 mL/min.
- Start the high-pressure pump.

#### 3 Observe the Eluent to cell connector

After a while, liquid must emerge from the **Eluent to cell** connector (wipe up fluid with paper towel).

If no liquid emerges at the **Eluent to cell** connector, then the preheating capillary is likely to be blocked. To eliminate the problem, see *Chapter Preheating capillary maintenance, page 28*.

#### 4 Observe the pump pressure

Observe the pump pressure display in the program part **Manual** of MagIC Net.

A constant pressure should establish itself after a while.

## 4.4 Testing the detector outlet capillary

The detector outlet capillary must be of a certain length in order to be able to generate sufficient backpressure. The required length is dependent on the flow that has been set. *The table 1* shows the recommended lengths, as determined by the set flow rate.

Table 1 Recommended lengths for the detector outlet capillary

Flow rate	Capillary length (□0.25 mm)
2.0 mL/min	0.5–1.5 m
0.5–1.0 mL/min	1.0–2.5 m
0.25 mL/min	3 m

To check whether the detector outlet capillary is free of blockages, proceed as follows:

### Testing the detector outlet capillary

Prerequisites:

- The detector inlet capillary is connected to the **Eluent in** connector.
- The high-pressure pump runs with a flow rate of 0.1 mL/min.

#### 1 Connecting the detector outlet capillary

Use a pressure screw (6.2744.014) to fasten the detector outlet capillary to the **Eluent to cell** connector.

#### 2 Adjusting the settings in MagIC Net

In the program part **Manual** of MagIC Net, increase the flow rate to 1.0 mL/min and wait until the pressure has stabilized.

#### 3 Observe the end of the detector outlet capillary

After a while, liquid must emerge from the end of the detector outlet capillary.

If no liquid emerges at the end of the detector outlet capillary, then the detector outlet capillary is blocked and must either be cut back once again or replaced.

**4 Loosening the detector outlet capillary**

Loosen the detector outlet capillary from the **Eluent to cell** connector. Wipe up emerging liquid with a cloth.

**5 Observe the pump pressure**

Observe the pump pressure display in the program part **Manual** of MagIC Net.

The drop in pressure should range from 0.1 MPa to a maximum of 0.3 MPa.

If the pressure differential is greater, then the detector outlet capillary must be cut back once again or replaced.

**6 Finish the test**

- In the program part **Manual** of MagIC Net, stop the high-pressure pump.
- Remove the detector outlet capillary from the **Eluent to cell** connector.

## 4.5 Testing the measuring cell

To test the measuring cell, proceed as follows:

### Testing the measuring cell

Prerequisites:

- The measuring cell is completely assembled (see measuring cell manual).
- The working electrode and the reference electrode are inserted (see measuring cell manual).

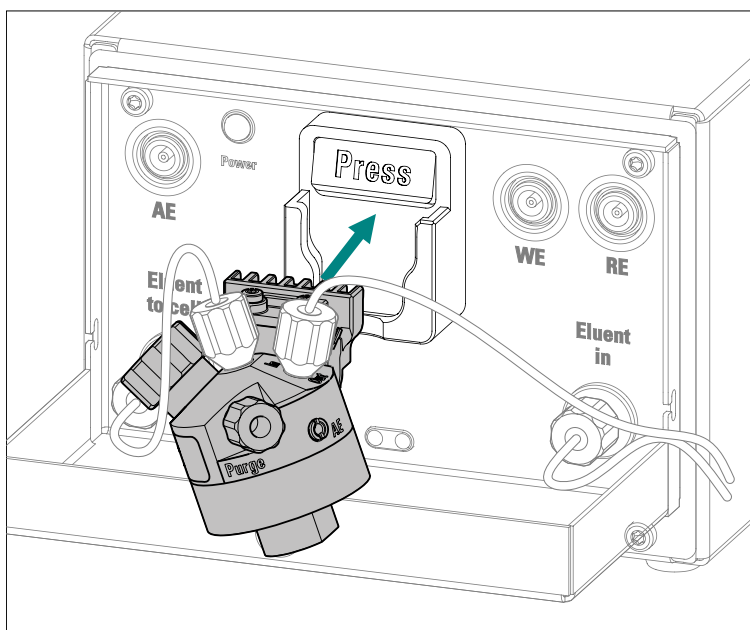
**1 Connecting the measuring cell**

- Connecting the measuring cell inlet:
  - *When the preheating capillary is used:* Use a pressure screw (6.2744.014) to fasten a piece of the PEEK capillary (6.1831.010) to the **Eluent to cell** connector on the detector.  
Use a pressure screw (6.2744.014) to fasten the other end to the **In** connector of the measuring cell.
  - *If the preheating capillary is not used:* Use a pressure screw (6.2744.014) to fasten the detector inlet capillary directly to the **In** connector on the measuring cell.

- Connecting the measuring cell outlet:  
Use a pressure screw (6.2744.014) to fasten the tested detector outlet capillary to the **Out** connector on the measuring cell (see "Testing the detector outlet capillary", page 20).

## 2 Inserting the measuring cell

Insert the chip of the measuring cell into the cell holder so that you can hear it locking in.



### NOTE

Do not move the measuring cell for at least 5 seconds after having inserted it.

During this time, data is read from the chip of the measuring cell and written into the database. This process must not be interrupted, because otherwise the data may be transferred incorrectly or incompletely.

## 3 Testing at low flow

- In the program part **Manual** of MagIC Net, set the flow rate of the high-pressure pump to 0.2 mL/min and start the high-pressure pump.

- Watch the detector outlet capillary: Liquid must emerge from the end of the detector outlet capillary.  
If no liquid emerges from the end of the detector outlet capillary:
  - Detach the capillary from the **Out** connection on the measuring cell and check whether the end has been pinched by the pressure screw.
  - Shorten the capillary and fasten once again to the **Out** connector on the measuring cell.
- Observe the measuring cell: No liquid should emerge from the body of the measuring cell.  
If the measuring cell is leaking:
  - Remove the measuring cell from the measuring cell holder.
  - Remove all capillaries and cables.
  - Check whether the pressure screw of the working electrode is properly connected and retighten it.
  - Restore the capillary connections.
  - Reinsert the electrode cables.
  - Reinsert the measuring cell.
  - Repeat the test.

#### 4 Testing at normal flow

- In the program part **Manual** of MagIC Net, raise the flow rate of the high-pressure pump to 1.0 mL/min.
- Observe the measuring cell: No liquid should emerge from the body of the measuring cell.

## 4.6 Deaerating the measuring cell

The cell must be deaerated in order to ensure that it contains no air bubbles.

The measuring cell must be deaerated after the installation and after each subsequent opening of the cell.

Proceed as follows:

### Deaerating the measuring cell

Prerequisites:

- The high-pressure pump is switched on and pumps the eluent through the IC system to the measuring cell.
- The measuring cell is switched off.

#### 1 Deaerating the reference electrode chamber

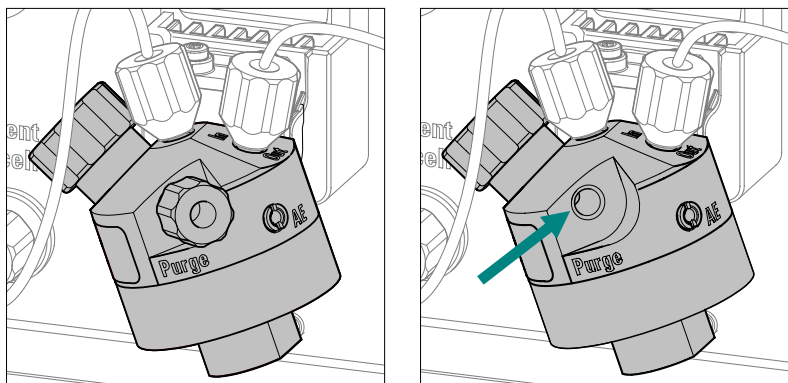
- Unscrew the nut on the RE connector and remove it.



- Lift out the reference electrode.
- Wait until the reference electrode chamber has filled with eluent.
- Reinsert the reference electrode. Wipe up any emerging eluent with a cloth.
- Screw the nut on the reference electrode connector back on tightly.

## 2 Removing the purge stopper

Remove the stopper from the **Purge** connector.



## 3 Deaerating the measuring cell

Observe the eluent that emerges through the deaeration opening. Wipe up liquid with a cloth.

Once no more air bubbles are visible, screw the stopper back on the **Purge** connector and tighten it by hand.

## 4 Switch off the high-pressure pump in MagIC Net.

# 4.7 Connecting the electrode cables



### CAUTION

The electrode cables may not be plugged or unplugged unless the measuring cell is switched off in the software.



### NOTE

The sockets and the plugs of the cables must be clean and dry.

### Connecting the electrode cables to the detector

Prerequisites:

- The measuring cell is switched off.
- 1** Plug the straight plug of the working electrode cable (red sleeve) into the **WE** socket of the detector.
  - 2** Plug the straight plug of the reference electrode cable (black sleeve) into the **RE** socket of the detector.
  - 3** Plug the straight plug of the auxiliary electrode cable (blue sleeve) into the **AE** socket of the detector.

### Connecting the electrode cables to the measuring cell

Prerequisites:

- The working electrode and the reference electrode are inserted into the measuring cell.
- 1** Plug the angled plug of the working electrode cable (labeled **WE**) into the working electrode socket.
  - 2** Plug the angled plug of the reference electrode cable (labeled **RE**) into the reference electrode socket.
  - 3** Plug the angled plug of the auxiliary electrode cable (labeled **AE**) into the socket (labeled **AE**).



## **4.8 Attaching the front cover**

In order to obtain good measuring results, we recommend that the front cover be put back in place.

When you are attaching the front cover, observe the following:

- Do not pinch any capillaries!  
Guide the capillaries through the capillary feed-throughs .
- Do not pinch any cables!

## 5 Operation and maintenance

### 5.1 General notes

#### 5.1.1 Care



#### WARNING

Untrained personnel may not open the instrument's housing.

The instrument requires appropriate care. Excess contamination of the instrument may result in malfunctions and a reduction in the service life of the sturdy mechanical and electronic components.



#### CAUTION

Even though design measures ensure that this will largely be prevented, the detector should be switched off without delay in the event that aggressive media have found their way into the interior of the detector. This is the only way to prevent extreme damage to the instrument electronics. In such cases, Metrohm Service must be informed.

Spilled chemicals and solvents should be removed immediately. In particular, the plug connections (particularly the power plug) should be protected from contamination.

Do not use scouring agents for cleaning the tray.

#### 5.1.2 Maintenance by Metrohm Service

Maintenance of the instrument is best carried out as part of annual service, which is performed by specialist personnel from Metrohm. A shorter maintenance interval is recommended if you frequently work with caustic and corrosive chemicals. Metrohm Service provides professional technical consultation at all times for the maintenance and servicing of all Metrohm instruments.

#### 5.1.3 Operation



#### CAUTION

In order to avoid disruptive temperature influences, the entire system must be protected from direct sunlight.

### 5.1.4 Shutting down

If the instrument is shut down for a longer period of time, the entire IC system must be rinsed as follows to rid it of salts in order to prevent eluent salts from forming crystals which may cause subsequent damage.

- Rinse all capillaries and the Dosino (if present) with methanol/ultrapure water (1:4).
- Rinse all pump tubings of the peristaltic pump with ultrapure water.

## 5.2 Amperometric detector

### 5.2.1 Maintenance



#### WARNING

When **rinsing the detector without column**, the pressure must not exceed **5 MPa**.

In order to ensure this, set the maximum pressure of the high pressure pump to **5 MPa** in MagIC Net.

### 5.2.2 Preheating capillary maintenance

The preheating capillary can become blocked, e.g. if the IC system has inadvertently been run dry.

To dissolve this blockage, proceed as follows:

#### Rinsing the preheating capillary

##### 1 Removing the separation column

Remove the separation column from the IC system and replace with a coupling (6.2744.040).

##### 2 Adjusting the settings in MagIC Net

In MagIC Net, adjust the following settings:

- Maximum pressure of the high-pressure pump: 5 MPa
- Flow rate: < 0.1 mL/min

##### 3 Rinse the system with the same eluent as before the blockage or with ultrapure water.

The eluent requires sufficient time to trickle through and dissolve the crystals.

- 4 Do not increase the flow rate until the pressure has stabilized.

If the preheating capillary remains blocked, then you can attempt to rinse the capillary in the opposite direction. To accomplish this, connect the detector inlet capillary to the connector **Eluent to cell** and repeat the procedure (see "*Rinsing the preheating capillary*", page 28).

If the blockage can also not be dissolved by rinsing in the opposite direction, then the preheating capillary must be replaced by a Metrohm Service employee.



<b>Problem</b>	<b>Cause</b>	<b>Remedy</b>
	<i>Measuring cell – Working electrode contaminated.</i>	Clean the working electrode (see the leaflet for the working electrode).
	<i>Measuring cell – Measuring cell leaking.</i>	Check the capillary connections on the measuring cell.
	<i>IC system – Eluent contaminated.</i>	Prepare a new eluent.
<b>Smooth baseline (no noise).</b>	<i>Communications problem between the amperometric detector and MagIC Net.</i>	<ul style="list-style-type: none"> <li>▪ Check whether the electrode cables are properly connected.</li> <li>▪ Check the electrode cable with dummy cell (see chapter 4.1, page 16).</li> <li>▪ Switch off the instrument, close and restart MagIC Net, switch the instrument back on.</li> </ul>
	<i>All of the data lies outside of the measuring range.</i>	<ul style="list-style-type: none"> <li>▪ Adjust the measuring range.</li> <li>▪ Deaerate the measuring cell (see "Deaerating the measuring cell", page 23).</li> </ul>
	<i>Short-circuit bridge between the electrodes.</i>	<ul style="list-style-type: none"> <li>▪ Examine the working electrode for prominent deposits.</li> <li>▪ Polish the working electrode (see the leaflet for the working electrode).</li> <li>▪ Replace the working electrode.</li> <li>▪ Clean the measuring cell.</li> <li>▪ Check the spacer.</li> </ul>
	<i>The reference electrode is worn out.</i>	Replace the reference electrode.
	<i>The cause is not clear.</i>	Perform a systematic error diagnostics (see chapter 6.9, page 37).
<b>The baseline has a large amount of noise.</b>	<i>Disruptive influences from outside.</i>	<ul style="list-style-type: none"> <li>▪ In the DC mode: Switch on the damping.</li> <li>▪ In the other measuring modes: Set a suitable smaller measuring range.</li> <li>▪ Attach the front cover.</li> </ul>
	<i>The Ag/AgCl reference electrode is worn out.</i>	Replace the reference electrode.
	<i>The auxiliary electrode is contaminated.</i>	Clean the auxiliary electrode of the measuring cell.
	<i>The working electrode is contaminated.</i>	<ul style="list-style-type: none"> <li>▪ Clean and polish the working electrode (see the leaflet for the working electrode).</li> </ul>



<b>Problem</b>	<b>Cause</b>	<b>Remedy</b>
		<ul style="list-style-type: none"> <li>Replace the GC working electrode if it has been used with oxidative potentials at the upper limit and polishing no longer helps.</li> </ul>
	<i>Air bubble in the measuring cell.</i>	Deaerate the measuring cell ( <i>see chapter 4.6, page 23</i> ).
	<i>The background current is too high, e.g. caused by contaminated eluent.</i>	Check the background current, e.g. use fresh eluent.
<b>The baseline is drifting.</b>	<i>IC system – Thermal equilibrium not yet attained.</i>	Condition the system with the heater switched on.
	<i>IC system – Leak in the system.</i>	Check the capillary connections and seal them.
	<i>IC system – Eluent is old (too much CO<sub>2</sub>).</i>	Prepare a new eluent.
<b>Unexpectedly high or low baseline.</b>	<i>Pd reference electrode – Working conditions not yet achieved.</i>	Equilibrate until the electrode has adjusted to the new elution conditions (over night).
	<i>DC method – Working conditions not yet achieved.</i>	An excessively high baseline is normal at the start of the equilibration. Equilibrate until the baseline corresponds to the one in the Application Works.
	<i>Detector parameters – Potentials set incorrectly.</i>	Set the potentials to correspond to the specifications in the leaflet and in the Application Works.
	<i>Incorrect eluent in the reference chamber.</i>	Remove the purge stopper on the measuring cell, wait until approx. 1 mL of eluent has emerged, screw the purge stopper back in tightly.
	<i>Electrodes contaminated.</i>	<ul style="list-style-type: none"> <li>Clean and polish the working electrode.</li> <li>Possibly clean the auxiliary electrode.</li> <li>Replace the reference electrode with a well-conditioned new reference electrode.</li> </ul>

## 6.3 General remarks regarding sensitivity fluctuations

Sensitivity fluctuations of up to 20% per week are normal for an unchanged system in constant operation.

The sensitivity can increase to approximately twice as much for a short time when new working electrodes are inserted or when the conditions change.

## 6.4 Problems with sensitivity

Problem	Cause	Remedy
<b>Declining sensitivity.</b>	<i>Measuring cell – Auxiliary electrode contaminated.</i>	Clean the auxiliary electrode (see measuring cell manual).
	<i>Incorrect eluent in the reference chamber.</i>	Remove the purge stopper on the measuring cell, wait until approx. 1 mL of eluent has emerged, screw the purge stopper back in tightly.
	<i>Sample concentration is no longer correct.</i>	Replace the sample and/or the standard solution.
	<i>Temperature fluctuations.</i>	<ul style="list-style-type: none"> <li>▪ Amperometric detector – Use preheating capillary.</li> <li>▪ IC instruments – Use column oven.</li> </ul>
	<i>Replace the measuring cell.</i>	<ul style="list-style-type: none"> <li>▪ Use a measuring cell of the same type.</li> <li>▪ Use the same spacer.</li> <li>▪ Use the same electrodes.</li> </ul>
	<i>Software – Measurement potential incorrect.</i>	Optimize the measurement potential.
	<i>Measuring cell – Working electrode contaminated.</i>	Clean the working electrode (see the leaflet for the working electrode).
	<i>IC system – Eluent contaminated.</i>	Prepare a new eluent.
	<i>IC system – pH of the eluent has changed.</i>	Check the pH value of the eluent and optimize it if necessary.



<b>Problem</b>	<b>Cause</b>	<b>Remedy</b>
<b>No measuring signal.</b>	<i>IC system – No power supply.</i>	Check the power connection and the supply voltage.
<b>Peaks cut off at the top.</b>	<i>Measuring range too small.</i>	<ul style="list-style-type: none"> <li>▪ Set a less sensitive measuring range.</li> <li>▪ Reduce the peak height, e.g. by means of sample dilution.</li> </ul>

## 6.7 Problems with the chromatogram

<b>Problem</b>	<b>Cause</b>	<b>Remedy</b>
<b>Peak drift with sugar analysis.</b>	<i>Carbonate absorption in the eluent.</i>	Use the Metrosep CO3 Trap 1 (6.1015.300) trap column.
<b>Peaks have poor resolution.</b>	<i>IC system – Diminished separating efficiency of the separation column.</i>	<ul style="list-style-type: none"> <li>▪ Regenerate the separation column (see <i>Chapter Operation and maintenance</i> in the manual for the IC instrument).</li> <li>▪ Replace the separation column (see <i>Chapter Operation and maintenance</i> in the manual for the IC instrument).</li> </ul>
	<i>IC system – Eluent is old.</i>	Prepare a new eluent.
	<i>The ionic strength of the sample or the pH value of the sample deviates greatly from the eluent.</i>	Dilute the sample or optimize the pH value of the sample.
<b>The retention times in the chromatogram have changed unexpectedly.</b>	<i>Absorption of analyte at the electrodes.</i>	Use a suitable combination of electrodes and eluent.
	<i>IC system – Diminished separating efficiency of the separation column.</i>	<ul style="list-style-type: none"> <li>▪ Regenerate the separation column (see <i>Chapter Operation and maintenance</i> in the manual for the IC instrument).</li> <li>▪ Replace the separation column (see <i>Chapter Operation and maintenance</i> in the manual for the IC instrument).</li> </ul>
	<i>IC system – Eluent is old.</i>	Prepare a new eluent.
	<i>The ionic strength of the sample or the pH value of the sample deviates greatly from the eluent.</i>	Dilute the sample or optimize the pH value of the sample.



<b>Problem</b>	<b>Cause</b>	<b>Remedy</b>
<b>Extreme spread of the peaks in the chromatogram. Splitting (dual peaks)</b>	<i>IC system – Dead volume at the ends of the separation column.</i>	Replace the separation column.
	<i>IC system – Dead volume in the IC system.</i>	Check the capillary connections.
	<i>Inhibition of the detection mechanism by the analyte (with PAD).</i>	<ul style="list-style-type: none"> <li>▪ Dilute the sample.</li> <li>▪ Allow the waveform to run-in better.</li> <li>▪ Adjust the PAD waveform.</li> </ul>
	<i>The column is overloaded.</i>	Dilute the sample.

## 6.8 Other problems

<b>Problem</b>	<b>Cause</b>	<b>Remedy</b>
<b>High background current.</b>	<i>IC system – Eluent contaminated.</i>	Prepare a new eluent.
	<i>Software – Measurement potential / pulse settings incorrect.</i>	Optimize the parameters.
	<i>Very wide peaks through substances with delayed elution.</i>	Wait for the complete elution of these substances.
<b>Unstable temperature.</b>	<i>The set temperature is too low.</i>	Set the temperature to at least 8 °C higher than the highest ambient temperature to be anticipated.
<b>Current display/ charge display in the software is frozen.</b>	<i>Measuring cell – electrodes are either not connected or not correctly connected.</i>	Connect the electrode connection cables correctly (see chapter 4.7, page 24).
	<i>Measuring cell – Small air bubbles in the measuring cell.</i>	Deaerate the measuring cell (see chapter 4.6, page 23).
	<i>Measuring cell – Electrode connection cable defective.</i>	Perform an instrument test with the dummy cell (see chapter 4.1, page 16).

## 6.9 Systematic error diagnostics

If the causes of a malfunction cannot be found among the problem descriptions in the above chapters, then proceed systematically as follows:

### Systematic error diagnostics

#### 1 Restarting the instrument and the software

- Switch off the instrument.
- Close and restart MagIC Net.
- Switch the instrument back on again.

If the problem has not yet been localized, continue with Step 2.

#### 2 Performing an instrument test with dummy cell

*(see chapter 4.1, page 16)*

If the problem has not yet been localized, continue with Step 3.

#### 3 Checking the software settings

- Check the method parameters of the detector and reset them to values that you know will function.
- Check the measuring range and reset it to values that you know will function or select a larger measuring range.
- Check manual changes to the settings and reset them to values that you know will function.
- Check manual settings in the time program and reset them to values that you know will function.

If the problem has not yet been localized, continue with Step 4.

#### 4 Cleaning the measuring cell

- Switch off the measuring cell.
- Remove the measuring cell.
- Clean the measuring cell (see measuring cell manual).
- Polish the working electrode (see the leaflet for the working electrode).
- Reinsert the measuring cell.

If the problem has not yet been localized, continue with Step 5.

#### 5 Replacing the reference electrode

If the problem has not yet been localized, continue with Step 6.



**6 Replacing the working electrode**

If the problem has not yet been localized, continue with Step 7.

**7 Replacing the body of the measuring cell**

Replace the body of the measuring cell with another one of the same type.

If the problem has not yet been localized, continue with Step 8.

**8 Requesting Metrohm Service**

If none of these measures help, please request Metrohm Service.



**NOTE**

Please note that, when the electrodes are replaced, the system requires a longer run-in time before the earlier values can be reproduced.

## 7 Technical specifications

### 7.1 Reference conditions

The technical specifications listed in this chapter refer to the following reference conditions:

<i>Ambient temperature</i>	+25 °C (± 3 °C)
<i>Instrument status</i>	Operating > 40 minutes (in equilibrium)

### 7.2 Leak sensor

<i>Type</i>	electronic, no calibration necessary
-------------	--------------------------------------

### 7.3 Ambient conditions

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



## 7.4 Energy supply

<i>Nominal voltage range</i>	100–240 V ( $\pm 10\%$ )
<i>Nominal frequency range</i>	50–60 Hz ( $\pm 3\%$ )
<i>Power consumption</i>	<ul style="list-style-type: none"> <li>▪ 65 W for typical analysis application</li> <li>▪ 25 W standby (conductivity detector to 40 °C)</li> </ul>
<i>Power supply unit</i>	<ul style="list-style-type: none"> <li>▪ up to 300 W maximum, electronically monitored</li> <li>▪ internal fuse 3.15 A</li> </ul>

## 7.5 Amperometric detector

<i>Type</i>	Microprocessor-controlled Digital Signal Processing (DSP technology)
<i>Potentiostat</i>	
<i>Potential range</i>	–5.0 to +5.0 V in steps of 0.001 V
<i>Potential step response time</i>	< 1 ms
<i>Detection modes</i>	<ul style="list-style-type: none"> <li>▪ DC</li> <li>▪ PAD</li> <li>▪ flexIPAD (flexible IPAD)</li> <li>▪ CV</li> </ul>
<i>Measuring unit</i>	
<i>AutoRange</i>	yes, (DC only)
<i>Digital signal range</i>	
<i>DC mode</i>	0.00012 pA–2 mA
<i>PAD mode</i>	0.012 pA–2 mA
<i>flexIPAD mode</i>	0.12 pC–200 $\mu$ C
<i>CV</i>	0.12 pA–20 mA
<i>Electronic noise</i>	
<i>DC mode</i>	< 2 pA
<i>PAD mode</i>	< 10 pA
<i>flexIPAD mode</i>	< 30 pC
<i>Filter</i>	
<i>DC mode</i>	Hardware filter, can be selected by the user
<i>All modes</i>	Software filter, can be set by the user

*Temperature control*

*Temperature stability at the heater* better than 0.05 °C with ambient temperature +8 °C–80 °C

*Operation*

*Direct* Via Software MagIC Net

*Remote* Via Remote Box

*Analog output* With 891 Professional Analog Out

*Output voltage* 0–1000 mV

*Full scale* Can be adjusted within the digital signal range

*Offset* Can be adjusted within the digital signal range

*System standby*

- Automatic function test at start-up
- Leak sensor
- Temperature stability monitoring

*Output channels*

- Current rating
- Charge

*GLP conformity*

Yes, optional

## 7.6 Housing

*Dimensions*

*Width* 365 mm

*Height* 131 mm

*Depth* 380 mm

*Material of housing*

Polyurethane hard foam (PUR) with flame retardation for fire class UL94V0, CFC-free, coated

*Controls*

*Indicators* LED for standby operation

*On/off switch* On the rear of the instrument



## 7.7 Interfaces

### USB

*Input* 1 USB upstream, type B (for connection to the PC)

*Output* 2 USB downstream, type A

*MSB* 2 MSB 8-pin mini-DIN (female) (for Dosino, stirrer, remote lines, etc.)

*Detector* 2 15-pin high density DSUB (female)

*Cell recognition* 1 on the front of the instrument

*Leak sensor* 1 jack plug

*Further connections*

- 1 15-pin DSUB (female)

## 8 Accessories

Up-to-date information on the scope of delivery and optional accessories for your product can be found on the Internet. You can download this information using the article number as follows:

### Downloading the accessories list

- 1** Enter <https://www.metrohm.com/> into your Internet browser.
- 2** Enter the article number (e.g. **2.945.0020**) into the search field.  
The search result is displayed.
- 3** Click on the product.  
Detailed information regarding the product is shown on various tabs.
- 4** On the **Included parts** tab, click on **Download the PDF**.  
The PDF file with the accessories data is created.



#### NOTE

Once you have received your new product, we recommend downloading the accessories list from the Internet, printing it out and keeping it together with the manual for reference purposes.



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